Virtual Lab Report of Department of Chemical Engineering



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Department of Chemical Engineering Contai Polytechnic This report is prepared as per Office order CP/E-1/2021:176 and CP/Virtual Lab/2021:208. This report is for the benefit of students studying diploma course in chemical engineering.

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SEMESTER-4th

ENERGY ENGINEERING LABORATORY

EXPERIMENT-1

To determine the Flash & Fire point of petroleum fraction

Introduction	Flash point is the lowest temperatures of the petroleum fraction, at which, vapor arising from the oil will ignite, flash, when exposed to a spark or flame under specific condition. Therefore, the flash point of fuel indicates the maximum temperature that it can be stored without serious fire hazards.
	Fire Point is the lowest temperature of the petroleum fraction, at which, vapor arising from the oil will ignite, i.e. fire, when exposed to a spark or flame under specific condition. Therefore, the fire point of fuel indicate the maximum temperature that its must not arrival to it to prevent the combustion of petroleum fraction.
Theory	A good lubricating oil reduces the loss of energy in the form of heat and it increases the efficiency of a machine. It should also not volatilize under the working temperatures. Even if the volatilization takes place to some extent, the vapors formed should not form inflammable mixtures with air under the conditions of lubrication. Therefore, the flash and fire point of a lubricating oil have a great importance. The flash point is defined as the minimum temperature at which the oil gives off sufficient vapor that ignite for a moment, when a flame is brought near it. While the fire point is the lowest temperature at which the vapors of the oil burn continuously for at least five seconds, when a flame is brought near it. Since a good lubricating oil should not volatilize under working conditions, so it should have flash point above the operating temperature in most cases. Similarly, in most cases, the fire points are 5 to 40 °F higher than the flash points. But these fire and flash points do not have any bearing with the lubricating property of the oil. This method is used to determine the flash point of lubrication oils and viscose materials. This method is not applicable to dry and solvent type liquid wax.
	The flash and fire points of lubricating oil may be determined by either open cup
	or closed cup apparatus.

First, we will discuss about the open cup apparatus, in which the oil is heated with its upper surface exposed to the atmosphere. Open and closed cup methods both can be used by Pensky Martens apparatus.

Objective	To determine the Flash Point & Fire Point of a given oil sample by Pensl	key
	Martens flash point apparatus.	
Procedure	 Clean the oil cup thoroughly and fill the oil cup with the sample oil to tested up to the mark. Insert the thermometer into the oil cup through a provision, wh measures the rise of oil temperature. Using the Energy regulator, control the power supply given to the hea and rate of heating. The oil is heated slowly when temperature of oil rises, it is checked for flash point for every 1°C rise in temperature. After determining the flash point, the heating shall be further continu The temperature at which time of flame application which causes burn for a period at least 5 seconds shall be recorded as the fire point. Repeat the experiment 2 or 3 times with fresh sample of the same oil. Take the average value of flash and fire points. 	be ich ater the led. ing
Observ ation Table	Sample Oil Flash Point, °C Fire Point, °C]
Calculations	The mean value of flash point is $\frac{1+2+3}{3} = \circ C$ The mean value of fire point is $\frac{1+2+3}{3} = \circ C$	
Results	 Time taken for flash point Time taken for fire point The flash point of the given oil sample (t1°C) The fire point of the given oil sample (t2°C) 	

Remarks	The flash point and fire point has been checked with the time successfully.
Lab Manual	https://web.iitd.ac.in/~pmvs/courses/mcl241/Flash%20and%20fire%20point.pdf
Experiment	https://www.youtube.com/watch?v=800OXkALzyo
al	
Graphics	
Relevant IS	
Code	
Quiz	Viva voce:
	1) What is Flash point and fire point?
	2) How do you calculate flash point and fire point of an oil sample?
	3) What is the flash point and fire point of petrol, diesel and kerosene?

SEMESTER-4th

ENERGY ENGINEERING LABORATORY



To determine the smoke point of kerosene

Introduction Theory	It is a measure of the tendency of a liqui soot. Generally, it is measured by burnir flame height is increased slowly till it star term of millimeters of smokeless flame smoke point. Hence, higher the smoke po smoke. Smoke point is related with the aromat proportional to the aromatic content. Smo tendency. Smoking tendency is proportion	d fuel to produce carbon particles known as ag fuel in a special wick lamp in which the ts producing smoke. The maximum height in at which flame starts smoking is termed as int, lower will be the tendency of the fuel to ic content of the liquid and it is inversely ke point is used to determination of smoking al to the aromatic content.
	The smoke point is the maximum flam burn without smoking in standard app smoke point of 22-25 mm. The smoking the test.	e height in mm to which a kerosene will paratus. Good quality of kerosene shows ng tendency of kerosene is determined by
	A - Too high B - Correct C - Too low Base of flame	Chamber Screen Flame Scale
	Fig.1 Typical flame appearance Good kerosene should be clean in respe on the wick smoking is due to aromatic provides an indication of the relative sm smoke point is related to the hydroo Generally, the more aromatic the produ high smoke point indicates a fuel of lo point is quantitatively related to the combustion products of the fuel.	Fig.2. Smoke point device ct to smoke in the flame and deposits char hydro-carbon primarily. This test method oke producing properties of kerosene. The carbon type composition of such fuels. ct is, the smokier it gets when it burns. A w smoke producing tendency. The smoke potential radiant heat transfer from the

	Candle Wick tube Wicks
	Figure 3: Smoke point apparatus Apparatus and component required: • Smoke point apparatus.
Objective Procedure	Kerosene To determine the smoke point and char value of kerosene
	 Soak a 125 mm long dried wick in the sample (kerosene) and place it in the wick tube of the candle. Fill the sample container (wick tube) up to desired level (10-20 ml) and introduce a wick in the container. Cut the wick horizontally from the end of the candle. Place the wick tube in the candle firmly and assemble in the burning chamber of the device. Open the glass door, light the flame and adjust the wick (The flame should be about 10mm height) and allow the lamp to burn for 5 min. Raise the candle until smoke appears from the chimney. Then lower the candle slowly through several stages until the smoke disappears. Take the reading from the reflection of the flame image on the scale. This reading represents smoke point of the sample. It is quite recommended that to take more than one observation to get right reading.

Observ ation Table	The smoke point in kerosene's are : Record of height in mm (h1) = Record of height in mm (h2) =
	Record of height in mm (h3) =
Calculations	The average smoke point of kerosene = $\frac{h1 + h2 + h3}{3}$
	=
Results	
	The average smoke point of kerosene is
	In this experiment the flame was yellow and the quality of kerosene is good.
Remarks	
	The smoke point has been checked three times successfully.
Lab Manual	https://koyapete.weebly.com/uploads/1/3/6/4/13645543/practical_oil_properties_expno. 1_smoke_point.pdf
Experiment	https://www.youtube.com/watch?v=Vm2kWOdmxsQ
al Graphics	
Relevant IS	
Code	
Quiz	Viva voce:
	2. What are the factors that affect measurement of smoke point?
	3. Why do we measure the smoke point of kerosene?
	4. How we can improve the smoke point of kerosene?

SEMESTER-4th

ENERGY ENGINEERING LABORATORY



To determine the Softening point of Bitumen

Introduction	The softening point is the temperature at which the substance attains particular degree of softening under specified condition of test. Bitumen does not suddenly change from solid to liquid state, but as the temperature increases, it gradually becomes softer until it flows readily. All semi-solid state bitumen grades need sufficient fluidity before they are used for application with the aggregate mix. For this purpose, bitumen is sometimes cut back with solvent like kerosene. The common procedure however is to liquefy the bitumen by heating.
Theory	The softening point is the temperature at which the substance attains particular degree of softening under specified condition of test. For bitumen, it is usually determined by Ring and Ball Test. A brass ring containing the test sample of bitumen is suspended in liquid like water or glycerin at a given temperature. A steel ball is placed upon the bitumen and liquid medium is then heated at a specified rate. The temperature at which the soften bitumen touches the metal plate placed at a specified distance below the ring is recorded as the softening point of a particular bitumen. It is obvious that harder grade bitumen possess higher softening point than softer grade bitumen. Apparatus : It consists of Ring and Ball apparatus. a) Steel Balls: They are two in number. Each has a diameter 9.5 mm and weights 2.5+0.5 gm b) Brass Rings: There are two rings of the following dimension: Depth : 6.4 mm
	 Inside diameter at bottom : 15.9mm Inside diameter at top : 17.5 mm Outside diameter : 20.6mm Brass rings are also placed with ball guides. c) Support: The metallic support is used for placing pair of ring. The upper surface of the rings is adjusted to be 50mm below the surface of water or liquid contained in the bath. A distance of 25 mm between the bottom of the rings and top surface of the bottom plate of support is provided. It has housing for suitable thermometer. d) Bath and Stirrer: A heat resistant glass container of 85 mm diameter and 120 mm depth is used. Bath liquid is water for materials having softening point above 80 °C, and glycerin for materials having softening point above 80 °C. Mechanical stirrer is used for ensuring uniform heat distribution at all times throughout the bath. Factors affect the test results: i. Quality and type of liquid ii. Weight of Balls iii. Distance between bottom of Ring and bottom base plate iv. Rate of heating

	Impurity in water or glycerin lies been observed to affect the result considerably. It is logical, lower will be the softening point, if the weight of balls is excessive. On the other hand, increased distance between bottom of ring and bottom plate, increases the softening point.
	Thermometer
	Softening Point Point Bitumen
	Strarting Point End Point
	Fig.1. Ring and Ball Apparatus
Objective	To determine the Softening point of Bitumen
Procedure	Sample material is heated to a temperature between 75 °C TO 100 °C above the approximate softening point until it is completely fluid and is poured in heated rings placed on metal plate. To avoid sticking of the bitumen to metal plate, coating is done to this with a solution of glycerin and dextrin. After cooling the rings in air for 30 minutes, the excess bitumen is trimmed and rings are placed in the support as discussed in item (c) above. At this time, the temperature of distilled water is kept at 5 °C. This temperature is maintained for 15 minutes after which the balls are placed in position. The temperature of water is raised at a uniform rate of 5 °C per minute with a controlled bottom plate by sinking of balls. At least two observations are made. For material whose softening point is above 80 °C. Glycerin is used in heating medium and the starting temperature is 35 °C instead of 5°C. The temperature at the instant when each of the ball and sample touches the bottom plate of support is recorded as softening point value. The mean of duplicate determinations is noted. It is essential that the mean value of the softening point (temperature) does not differ from individual observation by more than the following limits.

	 Record for each ring and ball, the temperature shown by the thermometer at the instant the sample surrounding the ball touches the bottom plate of the support, if any, or the bottom of the bath. Report to the nearest 0.5°C the mean of the temperature recorded m duplicate determinations, without correction for the emergent stem of the thermometer, as the softening point.
Observ ation Table	Number of balls Temperature
	1
	2
	Mean value
Calculations	
	The mean value of softening point of bitumen:
	1+2 = °C
	2
Results	
	The softening point of bitumen °C
Remarks	The determination of the Softening point of Bitumen has been checked successfully.
	 PRECAUTIONS The stirrer shall be placed in such a way that the moulds are not disturbed when the stirrer is in operation. The prescribed rate of heating shall be rigidly adhered to for ensuring accuracy of results. The rate of temperature rise shall not be averaged over the period of the test.
Lab Manual	http://www.darshan.ac.in/Upload/DIET/Documents/CI/2150601_Highway_Engineering_ Lab_Manual_11072018_025837PM.pdf
Experiment al Graphics	https://www.youtube.com/watch?v=-yBX14z70mI
Relevant IS Code	
Quiz	 Viva voce: 1. What is softening point? 2. What are the factors that affect measurement of softening point? 3. Why do we measure the softening point of bitumen?

COURSE-Diploma in Chemical Engineering SEMESTER-4th

ENERGY ENGINEERING LABORATORY



To determine the Thermal efficiency of kerosene stove

Introduction	
Introduction	 Kerosene is a product of crude oil and mainly consists of a mixture of hydrocarbons. In many developing countries, Kerosene is commonly used in urban households for both cooking and lighting. Kerosene stoves are widely available and affordable for urban population groups. Kerosene stoves are two types: Wick type stoves Pressurized stoves
Theory	
	Generally there is not much information available on kerosene stove tests or performance comparisons to other stove types. Simple stoves are constructed in the same way as oil lamps which use wick to draw fuel from the tank to the burner and which produce a lot of soot due to incomplete combustion. Improved kerosene wick stoves can have up to 30 or 40 wicks. A common design incorporates a series of wicks, usually made of loosely twisted or woven cotton, placed in a holder such that they can be moved up and down by a control knob. Wick stoves are cheap but also very unsafe. Here, the tank may heat up when used for a long time, causing an explosion of the stove. Pressurize stoves are more expensive, but also safer, more convenient and efficient. Air and kerosene can be premixed by means of hand pump. A pressurized kerosene fuel is passed under pressure through a nozzle and mixes with air to form a flame. To initiate the process the vaporizer has to be preheated using an alcohol based flame which burns for several minutes in a tray placed below the vaporizer. Once the temperature of the vaporizer is raised sufficiently the kerosene can then be vaporized by the heat of the cooking flame and the alcohol flame can be allowed to extinguish. The pressure forces kerosene through the vaporizer continuously and is controlled by the adjustment valve. To control the flame intensity , thus the heat output, the user can regulate the pressure of the tank by pumping the tank more to increase the pressure and make the flame larger , turning a small air screw (usually located in the filler cap) will release pressure from the tank and make the flame smaller.
	• <u>Advantages</u>
	1. On pressurize stoves cooking is fast.
	 Heat is easy to control Pressurize stoves may be very clean burning
	Disadvantages
	1. Kerosene is an exhaustible resource.
	2. When used in cheap wick stoves, kerosene can produce high levels of pollutants and contribute to indeer air pollution and dill health
	3. Wick type stoves can explode because the tank may heat up when used for
	a long time.
	4. Nozzles in pressurized cookers can get clogged by soot.

Objective	To determine the Thermal efficiency of kerosene stove
Observ	The kerosene stove is operated over a nominal range of three power settings — high, medium, and low — to heat water in two significantly different pot sizes (5 litres and 2 litres water). Features of the test protocol require a minimum of three tests under each condition (stove/fuel/pot combination) to obtain standard deviations and quality-assured generalizations about performance. Prior to each series of tests, fuels will be analysed for determination of their calorific values using a bomb calorimeter. The procedure is divided into three phases, a high power, a medium power and a low power test which were separately evaluated. The high power test begins with the pot, stove and water at room temperature. The stove is operated at its highest power setting until the water reaches a rolling boil. To obtain the specific fuel consumption at the high power setting, this test is continued for a minimum of 10 minutes after reaching the local boiling point. The medium power phase begins immediately after the first phase. The stove is adjusted to a medium power setting and allowed to reach a steady state (indicated by a constant rate of fuel consumption) usually for five minutes after which an identical second pot of cold water is placed on the stove. Mass, time and temperature readings are recorded while the water temperature is allowed to rise from ambient to ~70°C, marking the end of medium power test. The stove is next adjusted to a low power setting (or to the lowest level that provides a stable flame). An identical third pot of cold water is placed on the stove and data recorded until the water temperature has increased to ~70°C, marking the end of low power test. Note that the only cooking task performed was heating water. The thermal efficiency of kerosene stove at different power levels (high, medium and low) will be calculated.
ation	Mass of water in sauce pan =
	Change in water temperature =
	Mass of water evaporated from the pot =
	Mass of the fuel used =

Calcul ation	Thermal efficiency of kerosene stove —
	Overall efficiency = Total heat absorbed by water $\times 100\%$
	Total heat produced
	= Heat energy required to boil water + energy for vaporization \times 100%
	Heat energy released by given quantity of fuel
	$\eta = 4.186 \times m_{w} \times (T_{f} - T_{i}) + (M_{wv} \times h) \times 100\%$
	$F_{cd} \times LHV$ Where,
	$m_w = mass of water in pot$
	$(4.186 Jg^{-1} \circ C) =$ specific heat of water
	$(T_f - T_i) =$ change in water temperature
	M_{wv} = mass of water evaporated from the pot
	h = latent heat of evaporation of water = $2257 Jg^{-1}$
	F_{cd} = mass of the fuel used
	LHV = lower heating value of the fuel or net calorific value
Results	Thermal efficiency of kerosene stove =
Remarks	The determination of the Thermal efficiency of kerosene stove has been checked successfully.
Lab Manual	https://www.researchgate.net/publication/235732787_Performance_evaluation_and_emis sion_characterisation_of_three_kerosene_stoves_using_a_Heterogeneous_Stove_Testing Protocol_HTP
Experiment al Graphics	
Relevant IS Code	
Quiz	 Viva voce: 1. What is thermal efficiency? 2. How do you determine the thermal efficiency of a kerosene stove? 3. Write down the formula of thermal efficiency of a kerosene stove?

COURSE-<mark>Diploma in Chemical Engineering SEMESTER-</mark>4th

ENERGY ENGINEERING LABORATORY



To determine the Pour point & Cloud point of a Crude oil

Introduction	Cloud Point: Cloud point is the temperature expressed to the pearest degree centigrade
	at which oil becomes cloudy or hazy when oil is cooled under prescribed conditions.
	Pour Point: Pour point is the temperature at which oil ceases to flow. The pour point of a fuel is the lowest temperature at which it becomes semi solid and loses its flow characteristics.
Theory	Cloud point and pour points are related to low temperature characteristics of fuel and tells the behavior of fuel at low temperature. Haziness may be due to separation of waxes or increase in viscosity at low temperature. Due to the separation of waxes, viscosity of fuel oil increases and its fluidity decreases. To presence of solidified waxes thickens the oil and clogs fuels filters and injectors in engines. Usually the difference between cloud point and pour point is 4-6°F. Pour point tells us the temperature below which oil cannot be used as lubricant. Cloud point indicates the tendency of oil to plug filters or small orifices at cold operating temperatures. So, cloud and pour point also tell us the suitability of fuel oil in cold condition. The presence of some components in the formulation can depress or increase the cloud point of crude oil. For example, the addition of coupler or hydro trope can increase the cloud point temperature. Also, microfiltration can be used to separate waxes by precipitating out at different temperature in waxy crude oils.
Objective	To determine the Pour point & Cloud point of a Crude oil
	ro determine the rout point & Croud point of a Crude on

Procedure	Apparatus:				
	 Cloud & pour point apparatus Digital thermometer 				
	Cloud Point:				
	 Bring the sample to a temperature of at least 15 °C above the approximate cloud point and pour it into the jar to a height of 51 to 57 mm. Close the jar with the cork so that the thermometer bulb rests on the centre of the bottom of the jar. Fit the gasket on to the jar 25 mm from the bottom and insert the jar into jacket. Support the jacket and jar in a vertical position in the bath so that not more than 25 mm projects from the cooling medium. At each thermometer reading of one degree centigrade, remove the jar from the jacket quickly but without disturbing the oil, inspect the material for cloud, and replace the jar, this complete operation shall not take more than 3 sec. If the sample does not show a cloud when it has been cooled 10 °C. Place the jar and jacket in another bath maintained at a temperature of -15 °C to -18 °C. When an inspection of the sample first reveals a distinct cloudiness or haze at the bottom of the jar, record the reading of the thermometer as the cloud point after correcting the thermometer errors if necessary. 				
	Pour Point:				
 The sample has cooled enough to allow the formation of the crystals. Maintain the bath temperature at -1 °C to 2 °C Support the jacket and jar in a vertical position in the bath so that not me 25 mm projects from the cooling medium. Beginning at a temperature 12 °C above the expected pour point, thermometer reading which is a multiple of 3 °C, remove the jar from th carefully, and tilt it just enough to see whether the oil will move and the rest this complete operation shall not take more than 3 sec. As soon as the sample ceases to flow when the jar is tilted, hold th horizontal position for exactly 5 sec. If the sample shows any movement replace the jar in the jacket and coor the sample for another 3 °C. If the oil shows no movement during the 5 sec the reading of the thermometer. Add 3°C to the temperature recorded above and corrected for therm errors if necessary, and note down the result as the pour point. 					
observ ation Table	Sl. No.Cloud Point (°C)Pour Point (°C)				
1 AUIC					

Results	
	The cloud point of crude oil°C The pour point of crude oil°C
Remarks	 The determination of the cloud point and pour point of crude oil has been checked successfully. PRECAUTIONS: While observing the thermometer sometime there is a sudden rise or sudden drop in temperature. So, we should take care of it otherwise reading may alter. For pour point we must check the fluidity of oil for at least 5 second. Take care while observing cloud point it shows cloudy appearance in the oil while the cold condense water also shows cloudy appearance.
Lab Manual	https://www.bitswgl.ac.in/lab-manuals-mech/2.F&L-LAB-converted.pdf
Experiment al Graphics Relevant IS	https://www.youtube.com/watch?v=NQoxHavHJU0
Code	
Quiz	 Viva voce: 1. What is cloud point and significance of estimating cloud point? 2. What is pour point and significance of estimating pour point? 3. What are the properties of crude oil?

SEMESTER-4th

ENERGY ENGINEERING LABORATORY



To determine the proximate analysis of coal

Introduction	The proximate analysis of coal comprises of determination of the moisture, ash, volatile matter and fixed carbon.	
Theory	 Moisture Free water may exist in the coal as adsorbed on the surface, condensed inside fine capillary network and as bound to the coal molecule by chemisorptions and hydrogen bonding. Volatile Matter A volatile product obtained by the pyrolysis of coal in the absence of air is known as volatile matter. The product may contain hydrogen, methane, carbon monoxide, carbon dioxide, higher hydrocarbons, tar, water vapors, nitrogen, ammonia, hydrogen sulphide etc. The pyrolysis temperature of coal may lie in the range from 600-800 °C. The yield of volatile can be taken as a measure of its rank. Volatile matter will be much less in coke than that of coal as pyrolysis had occurred during coking at around 1000 °C. Volatile matter does not contain the moisture of coal but it contains water that is formed from the hydrogen and oxygen of coal during the decomposition. Ash Coals are associated with certain mineral or inorganic matter, which gets deposited into coal by subsequent infiltration. The ash consists mainly of silica, alumina, iron oxide and lime. When heated, coal as does not melt sharply at any temperature, but begins to soften at much lower temperature than that required for melting. The ash content in coke is much higher than in coal. Fixed carbon It measures the amount of non-volatile carbon remaining in a coal sample. Fixed carbon is obtained by deducting the sum of moisture ash and volatile matter 	
	percentage from 100.	
Objective	To determine the proximate analysis of coal.	
Procedure	 Moisture determination: (1) Dry the silica dish in an oven and measure its weight. (2) Spread out about 1 gm of 20 mesh coal sample on the dish. (3) Measure the weight of the dish again to find the exact mass of the sample. (4) Heat the dish without any cover in the oven at about 105±5 °C for 1 hour. 	

(5) Take out the dish from the oven, cover it with the lid and cool.

(6) Measure the weight of the dish to find the loss in weight of coal due to presence of moisture.



Volatile matter:

(1) Heat a clean crucible and its lid at 900±15°C for 7 minutes in muffle furnace.
 (2) Allow the crucible and lid to cool on a metal plate for a minute and in desiccators for 10 minutes.

(3) Measure the weight of the crucible and lid together.

(4) Put near 1 gm sample and measure the weight again to know the exact mass.

(5) Insert the crucible with the lid on it into the furnace at 900 $^{\circ}$ C and keep there for a period of 7 minutes.

(6) Measure the weight of the crucible with the lid to know the weight loss due to expulsion of volatile matter.



Ash determination:

(1) Dry the silica dish in an oven and measure its weight.

(2) Spread out about 1 gm of 20 mesh coal sample on the dish.

(3) Measure the weight of the dish again to find the exact mass of the sample.

(4) Insert the open dish in the furnace at 815°C for an hour.

(5) Remove the dish; allow it to cool for 10 minutes on the slab and 15 minutes in the desiccators.

(6) Measure the weight of the dish to find the mass left which is the ash content of the coal.



Observ					
ation Table	Sl. No.	Amount Moisture(gm)	Volatile Matter (gm)	Ash (gm)	Fixed carbon (gm)
1 ubic	Coal				—
					11
Calcul					
ation	% of Moi	sture = Loss in w	reight of coal samp	$le \times 100$	
		Weigl	ht of coal taken		
		=			
	% of Vola	atile Matter = Loss	s in weight of coal	sample at 925±20	0°C × 100
					Moisture
			Weight of coal	taken	
		=			
	% of Ash	= Weight of resid	ue left in crucible >	< 100	
		Weight of c	coal taken	-	
		=			
	% of fixe	d carbon = $100 - 9$	% of (moisture + vo	blatile matter + as	sh)
		=	× ·		, ,
Results					
	% of Moi	sture =			
	% of Vola	atile Matter =			
	% of Ash	=			
	% of fixed	d carbon=			
Remarks	The deter	rmination of prox	imate analysis of	coal has been c	checked successfully.

Lab Manual	https://www.iitk.ac.in/mme/test/MME331.pdf
Experiment al Graphics	https://www.youtube.com/watch?v=qu1v60L1Chk
Relevant IS Code	
Quiz	 Viva voce: 1. What is proximate analysis of coal? 2. How to measure the percentage of Ash? 3. How to measure the percentage of fixed carbon of coal?



ENERGY ENGINEERING LABORATORY

EXPERIMENT-7

To determine the calorific value of solid fuel

Introduction	Calorific value of fuel can be define as the amount of heat liberated when one unit mass or volume of the fuel undergoes completely combustion in presence of oxygen. When hydrogen is present in the fuel, it is converted to stream. High or gross calorific value (HCV or GCV): It is defined as the amount of heat evolves when one unit mass or volume of the fuel is completely burnt and combustible products are cooled to room temperature (25 °C or 77 °F). Low or net calorific value (LCV or NCV): It is defined as the amount of heat evolves when one unit mass or volume of the fuel is completely burnt and combustible products are permitted to escape. Therefore net calorific value is lower than gross calorific value.		
	Bomb calorimeter is used to find the calorific value of solid and liquid fuels.		
Theory Objective	Bomb calorimeter is used to determine calorific value of solid and liquid fuels experimentally. A bomb calorimeter contains a cylindrical bomb made by stainless steel. Combustion takes place in this cylinder. The lid contains two stainless steel electrodes. Oxygen is supplied through oxygen valve for combustion. The electrode is attached with a small ring which supports nickel or stainless steel made crucible. The bomb is taken in a copper calorimeter which is surrounded by air and water jacket in order to prevent heat loss by radiation. The copper calorimeter also contains electrically operated stirrer and Beckmann's thermometer (take reading with temperature difference up to 0.01°C).		
Objective	To determine the calorific value of solid fuel		

Procedure	 In a crucible, a known amount of the fuel is placed in the nickel or stainless steel crucible which is supported by a ring. A fine magnesium wire touches the fuel sample, which is already connected to the electrodes. The bomb lid is lightly screwed and filled with oxygen at about 25 atm pressure, is placed in copper calorimeter containing a known amount of water. The electrically operated stirrer is driven and notes the initial temperature of water (T1). After that both the electrodes are connected to a battery to complete the circuit. The fuel sample is burn and heat is liberated. To maintain the uniform temperature, water is continuously stirred and the final temperature (T2) of water is noted. Cotton Thread Correction: The heat liberated during the determination of calorific value, includes the heat given out by ignition of the fuse wire used. Hence, it must be subtracted from the total value. Acid Correction: The fuels containing S and N are oxidized, under high pressure and temperature of ignition, of sulphuric acid and nitric acid respectively. S + H₂ + 2O₂ → H₂SO₄ + Heat The above reactions are exothermic in nature. Thus, the measured amount of heat also includes the heat given out during the acid formation. Cooling Correction: The time taken to cool the water in copper calorimeter from maximum temperature to room temperature is recorded. From the rate of cooling and the actual time taken for cooling (t min), the cooling correction is added to the increase the temperature.
Observ ation	Mass of fuel (solid or liquid)= x g Mass of water taken = W g Water equivalent in g of calorimeter, stirrer, thermometer, bomb etc; = w g Initial temperature of water in calorimeter = T1 K Final temperature of water in calorimeter = T2 K

-

Calcul ation	So, Heat liberated by burning of fuel = $x L$		
	High or gross calorific value (L) = $(W+w)(T2-T1)$ cal/g		
	LCV = HCV - latent heat of water vapor		
	LCV = HCV – mass of hydrogen ×9× latent heat of steam (587 kcal/kg)		
	One part by mass of hydrogen produced nine parts by mass of water molecule. Theref		
	$LCV = HCV - H/100 \times 9 \times 587 \text{ kcal/kg}$		
	H = percentage of hydrogen in fuel		
	Corrections:		
	HCV or GCV = $(W+w)(T2-T1+Tc) - (Cs+Cn+Cf+Cc)$		
	Where		
	Tc = cooling corrections Cs = Corrections for sulphuric acid (H ₂ SO ₄) Cn = Corrections for nitric acid (HNO ₃) Cf = Corrections for fuse wire Cc = Corrections for cotton thread		
Results	High or gross calorific value = Low or net calorific value =		
Remarks	The determination of calorific value of solid fuel has been checked successfully.		
Lab Manual	https://www.lkouniv.ac.in/site/writereaddata/siteContent/202004132159500424ranvijay_ engg_Fuels.pdf		
Experiment al Graphics	https://www.youtube.com/watch?v=7vHz3YuMwbY		
Relevant IS Code			
Quiz	 Viva voce: 1. What is calorific value of a fuel? 2. Differentiate HCV from LCV. 3. Describe the method of determination of calorific value of a solid fuel by bomb calorimeter. 		

COURSE- Diploma in Chemical Engineering **SEMESTER**- 4th

HEAT TRANSFER LABORATORY

EXPERIMENT-1

To determine the linear expansion co-efficient of a Metal Rod

Introduction	The change in length per unit length per degree rise in temperature is called the coefficient of linear expansion. It is defined by $\Delta l = \alpha l \Delta T$ (Eq1) where Δl is the change in length, α is the average coefficient of linear expansion over the temperature change ΔT and l is the original length. Logically the initial temperature should be a fixed standard, such as 0 °C; however, because the value of <i>a</i> is very small for solids, the error introduced by using any other initial temperature is not large. The value of the coefficient of expansion should be specified as "per degree		
	centigrade" or "per degree Fahrenheit."		
Theory	In this experiment, a rod of an aluminum metal is encased in a metal jacket. Its length is measured at room temperature. The change in length is measured when the temperature is raised from room temperature to the temperature of steam. From these observations the coefficient of linear expansion will be computed for and compared with accepted values. Apparatus		
	Thermal expansion apparatus with thermocouple attached, steam generator, stop watch, digital thermocouple meter.		
Objective	Measure the coefficient of linear expansion of Aluminium Rod		
Procedure	In this experiment a dial gauge is used to measure the change in length Δl of an aluminium tube of length l. The insulated aluminium tube is heated by passing steam through it and its average temperature is measured by a thermocouple placed half way along the tube. The tube is fixed at one end and insulated from the base. The other end is free to move when the tube expands, this movement is measured by the dial gauge attached to the base.		
	 Measure the original length l of the aluminium tube. Fill the steam generator two-thirds full of water and turn it on and set the power to maximum. The steam enters the aluminium tube about ten minutes after the steam generator is switched on. Allow steam to flow through the jacket until a steady temperature is reached. Record the readings of the dial gauge and the thermometer. Turn off the water and drain the jacket. 		
Observation	Change in Temperature Change in Length (Δ l)		
Table			
Calculation	 From the difference in dial gauge readings, or the difference in scale readings, determine the change in length of the rod. From the initial and final temperatures, record the temperature difference. Calculate the coefficient of linear expansion by the use of Eq. (1). 		

	4) Compare this value with a standard value, taken over about the same			
	temperature range.			
	5) Compute the percent difference between the two, and discuss errors and			
	sources of errors.			
Remarks	• Make sure the steam outlet tube (at the dial gauge end) goes to the			
	sink.			
	• Steam can cause severe burns. Handle all hot apparatus with care			
Lab Manual	http://www.iiserpune.ac.in/~bhasbapat/phy221_files/lab1.pdf			
Experimental	Thermocouple			
Graphics	Dial t Meter			
	Fixed end &			
	Steam flow			
	Steam Free end & Insulation Thermocouple Aluminium			
	flow to steam outlet tube			
	\\↓ Staam			
	Generator			
Relevant IS				
Code				
Quiz	1. What is the unit of α ?			
	2. Does your value for the coefficient of linear expansion of aluminium			
	agree with the accepted value within the absolute error you have			
	3 What are the sources of error in this experiment? How would you			
	reduce these?			
HEAT TRANSFER LABORATORY

EXPERIMENT-2

Parallel Flow and Counter Flow Heat Exchanger

Introduction	Shell and Tube heat exchanger are popular in industries because they occupy less space and offered reasonable temperature drop. The apparatus consists of fabricated SS shell, inside which tubes with baffles on outer side are fitted. This is two pass heat exchanger so that hot water passes to one end of shell through the tubes and returns to another end through remaining tubes. The cold water is admitted at the one end of the shell, which passes over the hot water tubes. Valves are provided to control the flow rate of hot and cold water. Flow rates of hot and water are measured using Rota meters. A magnetic drive pump is used to circulate hot water from a recycle type water tank which is fitted with heaters and digital temperature controller.									
Theory	Heat exchangers are three types (I) transfer type, (ii) Storage type and (iii) Direct contact type. Transfer type heat exchangers are also three types (a) parallel flow type - in which fluids flow in the same direction. (b) Counter flow type - in which fluids flow in the opposite directions and (c) cross flow type - in which fluid flow at any angle to each other. A transfer type simple heat exchanger is taken into consideration in this experiment. One fluid flowing through the inner tube and the other through the annulus is surrounding it. The heat transfer takes place across the walls of the inner tube.									
Objective	To Find the Log	arithmic ient of S	Mean Tem hell and Tu	perature	Differer beat exch	nce (LM	TD), overa	ll heat		
Procedure	 Keep the Start the Start the Start the Adjust th Keep the Note the heat excl Repeat to counter the 	 Keep the thermometers in position. Start the flow on hot water side. Start the flow cold water side. Adjust the flow rate on the hot water side with the help of rotameter. Keep the flow rate same till the steady state condition is reached. Note the experiment for different flow rates for parallel as well as for counter heat exchanger. Repeat the experiment for different flow rates for parallel as well as for counter flow heat exchanger. 								
Observ ation	Type of		Hot Wate	r	0	Cold W	ater			
Table	Flow PARALLEL COUNTER	Mass flow kg/sec m _h	Inlet temp.°C T _{hi} , T ₂	Outlet temp. Tho, T ₁	Mass flow kg/se c m _c	Inlet temp T _{ci} T ₆ T ₄	Outlet temp.T _{co} T ₅ T ₃			



	i) Heat Transfer from Hot Water	Heat Transfer from Hot Water $q_1 = m_1 C_1 (T_1, T_2)$ Watt $m_2 = k\alpha/c_2 c_2$									
	$q_h = m_h C_h (1_{hi} - 1_{ho})$ Watt $m_h = kg/sec$ ii) Heat Transfer from Cold Water	$q_h - m_h C_h (1_{hi} - 1_{ho})$ watt $m_h = \kappa g/sec$ Heat Transfer from Cold Water									
	1) Heat Transfer from Cold Water $a_{1} = m_{1} C_{1} (T_{1} - T_{2})$ Watt $C_{2} = C_{1} = C_{2}$ of water	$a_{1} = m C_{1} (T_{1} - T_{2}) Watt C_{2} = C_{1} = C_{2} of water$									
	Heat Transfer rate, $q = UALMTD$	$q_c = m_c C_c (1_{co} - 1_{ci})$ watt $C_c - C_h - C_p$ of water Heat Transfer rate, $a = UALMTD$									
	LMTD = Logarithmic Mean Temperature Difference										
	$q = (q_h + q_i)/2,$										
	$T_{\rm m} = -\frac{\Delta T_i - \Delta T_0}{ln(\frac{\Delta T_i}{\Delta T_0})}$										
	Where, $\Delta T_i = \text{Temp. difference at inlet.}$ $\Delta T_0 = \text{Temp. difference at outlet.}$ q = A.U.LMTD										
	<i>q</i>										
	$U = \frac{1}{A(LMTD)} W/m^{2} C$										
	A = Surface Area = $\pi DL m^2$										
	Calculation Table:										
	$LMTD(T_m)$ O U										
	$(^{\circ}C)$ (W) $(W/$										
Remarks	• It is recommended to open the control valves followed by activating th	e									
	pumps • After changing the flow rote or flow directions, operator should writ for										
	• After changing the now-rate of now-directions, operator should wait to 10/15 minutes to reach steady state	1									
	• Never run the pump dry										
	• Wear heat-resistant gloves before operating the hot water valves.										
Lab Manual	https://www.che.utah.edu/site-specific-resources/chemical-										
	engineering/department_equipment/Projects_Lab/H_Double_Pipe_Heat_Exchanger	/									
	MIS Report - Double Pipe.pdf										



HEAT TRANSFER LABORATORY

EXPERIMENT-3

To determine the overall heat transfer co-efficient for a shell and tube heat exchanger

A Shell and Tube heat exchanger is the most widely used heat exchange equipment. This Introduc tion type of heat exchanger consists of a number of tubes in parallel enclosed in a cylindrical shell. Heat is transferred between one fluid flowing through the tube bundle and the other fluid flowing through the cylindrical shell around the tubes. Shell and Tube heat exchanger are popular in industries because they occupy less space and offered reasonable temperature drop. The apparatus consists of fabricated SS shell, inside which tubes with baffles on outer side are fitted. Shell-and-tube heat exchangers can have multiple passes, such as 1-1, 1-2, 1-4, 1-6, and 1-8 exchangers, where the first number denotes the number of the shells and the second number denotes the number of passes. The schematic diagram of 1-1 pass is shown below. Tube outlet Shell inlet Shell Tube Shell sheet Baffles End channel Shell outlet Tube inlet 0 0 0 0 0 0 000000 00000 0 0 00 Figure: Schematic of one-shell one-pass (1-1) shell-and-tube heat exchanger. A heat exchanger is a device used for effectively carrying out the process of heat Theory exchange between two fluids that are at two different temperatures. The heat exchangers are useful in many engineering processes like those in refrigerating and air conditioning systems, power systems, food processing systems, chemical reactors and space or aeronautical applications. Common examples of Heat exchanger are the radiator of a car, the condenser of the back of the domestic refrigerator, the steam boiler of a thermal power plant. Heat exchanger can be classified in three categories: 1. Transferred Type 2. Storage Type 3. Direct Contact Type A Transferred Type heat exchanger is one in which both fluid passes simultaneously through the device and the heat is transferred through separating walls. In practice most of the heat exchangers used are transferred type one. The transferred type heat exchangers are further classified according to the flow arrangement as 1. Parallel Flow: In which fluids flow in the same direction 2. Counter Flow: In which they flow in opposite direction 3. Cross Flow: In which they flow at right angles to each other. Shell and Tube Heat Exchanger: In shell and tube heat exchanger, the fluid flowing through the tubes is called the tube

	fluid whereas the fluid flowing outside the tubes are called the shell fluid. Depending
	upon the heat transfer area requirement we can have multi tubes and or shell pass.
	The flow conditions for shell and tube type heat exchanger are neither parallel flow nor
	counter flow type. To create turbulence in the shell side fluid and enhance the cross-flow
	velocity of this fluid relative to the tubes, haffles are generally provided. This results in a
	velocity of this field relative to the tubes, barnes are generally provided. This results in a
	nigher heat transfer coefficient for the outer tube surface.
	When large quantities of heat are to be transferred the heat transfer area requirement of
	heat exchanger also becomes large. In a single pass heat exchanger this requirement can
	be met either by increasing length, of tubes or by decreasing the diameter and increases
	the nos of tubes at the same time. Neither these methods are practical because due to
	limitations of sides the length of tube cannot be increased arbitrarily and large pressure
	drame early economy with smaller dismeter types. These difficulties lead us to multi ness
	drops could occur with smaller diameter tubes. These difficulties lead us to multi pass
	arrangement.
	Apparatus description:
	Apparatus description.
	The laboratory experimental set-up is a two-pass heat exchanger, so that hot water passes
	to one end of shell through the tubes and returns to another end through remaining tubes.
	The cold water is admitted at the one end of the shell, which passes over the hot water
	tubes. Values are provided to control the flow rate of hot and cold water. Flow rates of
	hat and cold water are massured using Date maters. A magnetic drive nump is used to
	not and cold water are measured using Rota meters. A magnetic drive pump is used to
	circulate hot water from a recycle type water tank which is fitted with heaters and digital
	temperature controller.
	Logarithmic Mean Temperature Difference (LMTD)
	Logaritanie Aroan Femperature Difference (EATED)
	For the derivation of temperature difference of two fluids, the following assumptions
	must be made:
	1. The overall coefficient of heat transfer U is constant over the entire length of path.
	2. The fluid flow is constant obeying the steady state requirement
	3. The specific heat is constant over the entire length of path.
	4. There are no partial phase changes in the system i.e. vaporization or condensation. The
	derivation is the application for the sensible heat changes and when vaporization or
	condensation is isothermal over the whole length of path.
	5. The heat loss is negligible
Objectiv	To determine the overall heat transfer co-efficient for a shell and tube heat exchanger
e	
Procedu	STARTING PROCEDURE
re	1. Clean the apparatus and make the water bath free from dust.
	2. Close the entire drain valve provided.
	3. Fill the water in the bath and switch on the heater.
	4. Adjust the required temperature of hot water.
	5. Adjust the valve. Allow the hot water to recycle bath through by pass
	6. Switching on the magnetic pump.
	/. Start the flow through the shell and run the exchanger.
	A dust the flow rate on cold water side with the help of Rotameter.

	9. A 10. K 11. R	ljust the flow rate eeping the flow rate ecord the temperat	on hot wat tes same, v ures on ho	ter side w vait till ste t water ar	ith the he eady state id cold-w	elp of Rota e condition vater side	ameter. ns are reache accurately.	d.
	CLOS 1. Wh 2. Sw 3. Sw 4. Dra	SING PROCEDU en experiment is c itch off magnetic p itch off power sup in water bath with	RE over switch pump for h ply to the p help of dr	n off the h ot water s panel. rain valve	eater firs supply s.	:t.	-	
Ob ser	S	Hot	Water			Cold Wat	er	
vat	No	·	·· ater					-
ion		Mass flow rate I PH	Inlet	Outlet	Mass	Inlet		
Ta ble		m _h	temp.°C	temp.	rate LPH	temp.	Outlet tempºC	
bic			T_1	T ₂	mc	T ₃	T4	
	1.							
Calcelet	2.							
	Data Inner Outer lengtl n= no iii iv	Given: Tube - Dia 0.013 Tube - Dia 0.013 Tube - Dia 0.016 a 0.5 m of tubes) Heat Transfer $q_h = m_h C_h($) Heat Transfer LMTD = L $q = (q_h + q_c)/$ $T_m = \frac{\Delta T_1 - \Delta}{ln(\frac{\Delta T_2}{\Delta T_2})}$ Where, Δ Q Overall Heat to $U = \frac{q}{A_0(LMTD)}$ $A_0 = Surface \Delta$	f m f m f m f from Hor T _{hi} - T _{ho}) V f from Cor T _{co} - T _{ci}) ' rate, q = U ogarithmic 2, $\frac{T_2}{\frac{1}{2}}$ T ₁ = Temp T ₂ = Temp. = A.U.LM transfer coe $\frac{1}{2}$ W/m ² °C Area = n π D	t Water Watt $m_h=$ old Water Watt $C_c =$ ALMTD Mean Ten 0. different MTD fficient,	kg/sec $C_h = C_p$ nperature ace at inleve at outl	of water Difference et. et.		

		Calculation T	able:			
		Sl. No.	LMTD (T _m) (°C)	Q (W)	U (W/m ^{2°} C)]
			()	(**)		
	-					
Remark s	•	It is recommend After changing	led to open the co the flow-rate or	ntrol valves follo flow-directions,	wed by activating the operator should wait	pumps for 10/15
	•	Never switch switches given	on mains power on the panel are a	supply before t OFF position.	ensuring that al the	ON/OFF
	•	Never run the p Wear heat-resis	ump dry tant gloves before	operating the ho	ot water valves.	
	•	Always keep th	e apparatus free fi	rom dust.		
Lab Manual	https://w _ENGR	www.academia. 3930U_Heat_7	edu/30188162/EN Transfer_Experime	ent_4_Shell_and	ABORATORY_MAN _Tube_Heat_Exchang	VUAL_for
Experim ental Graphic s				Shall		
			Connections Tubesheet Gask Head	Shell Baffles Mounting	ube undle	

	Shell Dutlet Tube Inlet Tube Inlet Tube Inlet Tube Dutlet Tube Dutlet Thermocauple
	https://www.pdhonline.com/courses/m371/m371content.pdf
Releva nt IS Code	
Quiz	 Why LMTD is used in heat Exchangers? Why baffles are used in the Shell and Tube type Heat Exchangers? In a shell and tube Heat Exchanger, if we want to heat a stream of liquid by Steam, then in which side steam and liquid should be kept, respectively?

HEAT TRANSFER LABORATORY

EXPERIMENT-4

To determine rate of evaporation in a jacketed open pan evaporator

Introduction	In the evaporation process, concentration of a product is accomplished by boiling out a solvent, generally water. The recovered end product should have an optimum solids content consistent with desired product quality and operating economics. It is a unit operation that is used extensively in processing foods, chemicals, pharmaceuticals, fruit juices, dairy products, paper and pulp, and both malt and grain beverages. Also it is a unit operation which, with the possible exception of distillation, is the most energy intensive.
Theory	Open Pan Evaporator is a device for concentrating a solution by vaporizing part or all of the solvent. In most of the cases the solvent is water. The pan evaporator set-up is designed to study the fundamentals of evaporation process. The set-up consists of a jacketed pan evaporator made of stainless steel and an electrically heated steam generator of suitable capacity. To evaporate the solution in pan, steam is allowed to enter in the jacket using a control valve. Condensate is collected from steam trap for energy measurement. Heat transfer areas normally are quite small due to vessel shapes and heat transfer coefficients (HTC's) tend to be low under natural convection conditions. Low surface areas together with low HTC's generally limit the evaporation capacity of such a system. Heat transfer is improved by agitation within the vessel. In the open pan evaporator, steam surrounded the flask containing liquid water. According to heat transfer laws, the energy in the steam will transfer to the water causing some component of liquid to vaporize into gas (steam). The newly formed steam (distillate) is collected. In an open pan evaporator is defined as the no of kilograms of water evaporated per hour. Capacity of an evaporator is defined as the no of kilograms of water evaporated per hour.

	m _s is the rate of steam flow								
	λ_s is the latent heat of vaporization								
	m is mass of liquid evaporated								
	λ_v is the latent of evaporation at boiling point								
	m_f is the mass of solution								
	H is the enthalpy of liquid at boiling point								
	H _f is the enthalpy of feed liquid								
Objective	To determine rate of evaporation in a jacketed open pan evaporator.								
Procedure	1. Clean the pan and find its weight W_1 kg.								
	2. Prepare a 10-20% NaCl solution.								
	3. Transfer the solution to the pan.								
	4. Take the weight of the solution with pan W_2 kg.								
	5. Heat the solution using steam through the jacket for 1 h.								
	6. Switch off the heater.								
	7. Allow the solution to cool.								
	8. Note down the weight of the solution W_3 kg.								
Observation	Initial weight of non W - ka								
UDSERVATION Table	$W_{i} = \frac{1}{2} \sum_{k=1}^{n} \frac{1}{2} \sum_{k=1}^$								
Table	Weight of pan and diffue solution $w_2 = \dots \dots kg$								
	Weight of pan and concentrated solution $w_3 = \dots kg$								
	Weight of water evaporated = W_2 - W_3 =kg								
Calculation	Capacity of evaporator =								
Demonstra	There should not be any lookes of water from the non								
Lah Manual	I here should not be any leakage of water from the pan.								
Experimental	1111								
Experimental Graphics									
Relevant IS									
Code									
Quiz	Define evaporation								
	• Define capacity and economy of evaporator								

HEAT TRANSFER LABORATORY

EXPERIMENT-5

Determination of Thermal Conductivity of Metal Rod

Introduction	Thermal Conductivity is the physical property of the material denoting the case
	with a particular substance can accomplish the transmission of thermal energy
	by molecular motion
Theory	Thermal energy can be conducted in solids by free electrons and by lattice
	vibrations. For a homogeneous solid material, it may be defined from the
	Fourier Law of heat conduction as amount of heat conducted per unit time
	through unit area normal to the flow direction, when a temperature difference
	of unit degree is maintained across unit thickness.
	Thermal conductivity $K = -adI/AdT$ (1)
	Where, $q =$ Heat conducted, watts
	A = Area of conduction heat transfer, m^2
	dL = distance over which temperature difference need to be measured, m
	dI = temperature difference across the length dL, °C K = Thermal Conductivity, Watt/m°C
Objective	To determine the thermal conductivity of a good conducting material (e.g.
Objective	Brass)
Procedure	
	i. A brass rod one end of which is heated by an electric heating coil,
	while the other end is inside the cooling water jacket.
	ii. The middle portion of rod is surrounded by an insulating material to
	minimize lateral heat transfer from rod and thus ensure a more nearly
	constant temperature gradient throughout the length of the rod.
	iii. The temperature of the bar is measured at five different sections.
	iv. Water is circulated through the jacket and its flow rate and temperature
	rise can be noted down.
	v. The neater is provided with a dimmerstat for controlling the neat input.
	vi. Digital indicators for voltage, current and temperature.
	Steps:
	1 Switch on the dimmer and give some low input
	2. The difference of temperature should not be more than 4-6 °C for water
	inlet and outlet.
	3. Open the inlet cock full. Outlet valve should be opened such a way that
	water should come drop by drop.
	4. Allow the unit for stabilization (Temperature indicator should read
	5 Note down the readings: collect the water in a measuring cylinder
	6 Repeat the experiment for different inputs
	o. Repeat the experiment for different inputs.
	PHYSICAL DATA:
	Length of metal $Bar = 460 \text{ mm}$
	Diameter of metal Bar = 20 mm
	Test length of the bar, dL=200mm
	No. of thermocouples mounted on the bar $= 5$
	No. of thermowells in the water line $=2$
	Cooling water Jacket dia =90 mm
	Temperature indicator = $0-300 \text{ °C}$

	Dimmar stast for water coil = 1.5 KW															
	Length of metal bar between 1 and $5 = 200 \text{ mm} = 0.2 \text{ m}$															
Observation	Sl Heat Input W= Thermocouple Reading (°C) Water Mass										Mass					
Table	No.	No. V* I Temperature flow														
																rate, Kg/s
		V (v)			T ₁	T ₂	T ₃	T ₄	T ₅	T ₆	T ₇	T ₈	T9	T ₁₀	T ₁₁	
		(')	(11)	(**)												
Calculation	Plot 1	the v	ariat	ion o	f tem	nera	ture	alone	o the	test	leng	th of	`the 1	rod K	σ/s	
Calculation	From	the the	grap	h. ob	tain o	dT/d	L. w	hich	is the	e sloi	be of	the	strai	ght lin	e pass	ing
	throu	igh/n	lear t	o the	poin	ts in	the g	graph	1.]				8	- F	8
	Assu	ming	g no l	heat l	oss,			- 1								
	Heat	cond	ducte	d thr	ough	the	rod =	= Hea	at car	ried	awa	y by	the c	cooling	g wate	r.
	-KAG	dT/d	L = 0	Qv p _w	Ср	(T ₁₁ -	T10)									
	$A = \pi$:D ² /4	•, m²	.:. a			· · · · · ·		3/-							
	QV = Cn =	= voll	imet	ric iic beat	of w	ite oi	= 1 2	er, m	r/S	r						
	$p_{m}=0$	- spe densi	ity of	f wate	or w •r = 1		– 4.2 ko/n	. кл/к n ³	.g. (_						
	Thus	. K =	= Ov	$p_w C$	n (T ₁	1-T1	a)dL	/(A d	T)							
Remarks	After	r star	ting	the ex	kperi	men	t, wa	it for	atle	ast 1	5 mi	n foi	reac	hing s	steady	state
Lab Manual		httj	os://s	ites.g	googl	e.coi	n/vie	ew/v]	lab-b	nmit	mec	h/ho	me/h	eat-tra	ansfer-	
]	lab/d	leterr	ninat	ion-c	of-the	erma	l-con	duct	ivity	-of-a	ı-me	tal-ro	od?aut	huser=	=0
Experimental	Sche	mati	c Dia	ıgram	1 :											
Graphics																7
			-0	r			_					-				
						-									-	
			5	27 L		17	F- 50		-	Sümm	-	-	4	WATERO	UT	
		-			-	Streen	1	50	mm L	-			LWATE	RIN		
			1257 13	L			-		460 mm					_		
	*															
	Refe	rence	e Det	ails:												_
	1	н	eater	250	watte	2										
	2.	St	vecin	nen (1	Brass	s, rođ) 20 -	mm 1	Dia	460 1	mm	Long	ŗ			
	3.	G	lass	Wool	as a	n Ins	ulato	or,	_ 1u,			_ 0112	,			
	4.	W	ater	Cool	ing J	acke	t wit	h Ins	ulati	on						
	5.	D	imm	er for	Hea	t Co	ntrol	ler,								
	6.	D	igital	l Volt	mete	er,										

	. Digital Temperature Indicator,							
	8. Digital Ammeter,							
	Thermocouple Selector Switch,							
	0. M.S. Shell,							
	11. Panel Board							
	Thermocouple Details:							
	$T_1 - T_5 =$ Temp along the length of test specimen							
	$T_6 - T_9 = MS Body Temperature (Shell)$							
	$T_{10} =$ Water Inlet Temperature							
	$T_{11} =$ Water Outlet Temperature							
	1							
Relevant IS								
Code								
Quiz	4. Define thermal conductivity.							
	5. Why are the most metals good conductor of heat?							
	6. How will you determine thermal conductivity of a cylinder?							

HEAT TRANSFER LABORATORY

EXPERIMENT-6

To determine the thermal conductivity of bricks in series.

	Area of brick = mm
	Thickness of brick T ₁ , T ₂ , T ₃ are known
	Thermal conductivity of materials $K_1 = K_2 = K_3 = K$ which is known
	Considering the composite slab made up of three materials of
	thickness L ₁ , L ₂ , and L ₃ having thermal conductivities K ₁ , K ₂ and K ₃
	heat transfer can be calculated using Fourier's Law as
Observation	$\begin{vmatrix} \mathbf{SL} & \mathbf{V} & \mathbf{I} & \mathbf{VI} & \mathbf{T}_1 & \mathbf{T}_2 & \mathbf{T}_3 & \mathbf{T}_4 & \mathbf{T}_5 & \mathbf{T}_6 & \mathbf{T}_7 & \mathbf{T}_8 \\ \mathbf{No} & (\mathbf{V}) & (\mathbf{A}) & (\mathbf{W}) & \mathbf{V} & \mathbf{V} \end{vmatrix}$
Table	
Calculation	At steady state the rate of heat flow through the individual layers are equal
	So we can write the following set;
	$q/A = K_1/L_1 [(T_1+T_2)/2 - (T_3+T_4)/2]$
	$q/A = K_2/L_2 [(T_3+T_4)/2 - (T_5+T_6)/2]$
	$q/A = K_3/L_3 [(T_5+T_6)/2 - (T_7+T_8)/2]$
	$T_1 \& T_2 =$ Interface temperature of 1 st brick and heater, °C
	$T_3 \& T_4 =$ Interface temperature of 1 st and 2 nd brick, °C
	T ₅ & T ₆ = Interface temperature of 2^{nd} and 3^{rd} brick, °C
	$T_7 \& T_8 = Top surface temperature of 3rd brick, °C$
	q = heat input to the system,
	Overall temperature difference $\Delta T = [(T_1-T_7)+(T_2-T_8)]/2$
	Total thickness of wall, $\Delta L = L_1 + L_2 + L_3$
	Thermal Conductivity of composite wall $K_{eff} = q\Delta L/\Delta T W/m^{\circ}C$
	$\Delta I = (1_1 + 1_2)/2 - (1_7 + 1_8)/2$ K = Thermal conductivity of comparison with multi W/m0C
	$K_{eff} = 1$ nermal conductivity of composite wall, w/m ⁻ C
Domorks	. Koon all the assembly year distynhed
KUIIAI KS	Reep all the assembly undisturbed.
	• Remove air gap between plates by moving nand press gently.
I ah Maaaal	• Operate selector switch of temperature indicator genuy.
Lab Manual	Nil Nil
Experimental	Schematic Diagram:
Graphics	
	A Designed and the second s
Relevant IS	
Code	
Quiz	7. Define thermal conductivity.
	8. How will you determine thermal conductivity of a rectangular slab?

HEAT TRANSFER LABORATORY

EXPERIMENT-7

To determine the rate of heat transfer through bricks in series

Introduction	In engineering applications, we deal with many problems. Heat Transfer
	through composite walls is one of them. It is the transport of energy between
	example, a fastener joining two mediums also acts as one of the layers between
	these mediums. Hence, the thermal conductivity of the fastener is also very
	much necessary in determining the overall heat transfer through the medium.
	An attempt has been made to show the concept of heat transfers through
	composite walls.
Theory	A direct application of Fourier's law is the plane wall. Fourier's equation applying
	$a = -K_1 \Delta (T_2 - T_1)/L_1$
	$q = -K_2 A (T_3 - T_2)/L_2$
	$q = -K_1A(T_4-T_3)/L_{3}$
	Where, $q =$ Heat conducted, watts
	A = Area of conduction heat transfer, m^2 L x = distance over which temperature difference need to be measured m
	ΔT = temperature difference across the length dL, °C
	K = Thermal conductivity, Watt/m°C
	The Apparatus consists of a heater sandwiched between two asbestos sheets
	Three slabs of bricks are provided on both sides of heater, which forms a
	composite structure. A small press- frame is provided to ensure the perfect
	contact between the slabs. A variac is provided for varying the input to the
	Digital Ammeter Temperatures Sensors are embedded between inter faces of
	the slab, to read the temperature at the surface. The experiment can be
	conducted at various values of power input and calculations can be made
	accordingly. End losses from the composite wall are minimized by providing thick insulation all rounds to ansure unidiractional heat flow.
	thick insulation all founds to ensure undirectional near now.
Objective	To determine the rate of heat transfer through bricks in series
Duosoduuo	we Ensure that Mains ON/OFF switch siver on the neural is at OFF
rocedure	xv. Ensure that Mains ON/OFF switch given on the panel is at OFF position & dimmer-stat is at zero position
	xvi. Connect electric supply to the set up.
	xvii. Switch ON the Mains ON / OFF switch.
	xviii. Set the heater input by the dimmer-stat, voltmeter in the range 40 to 100 V.
	xix. Note down the reading of voltmeter, ampere meter and temperature
	sensors in the observation table
	xx. After experiment is over set the dimmer stat to zero position.
	xxi. Switch OFF the Mains ON/OFF switches.
	AAn. Switch Off cloude supply to the set up.
	DIVELCAL DATA.
	THI SICAL DATA.
	Area of brick = mm
	Thickness of brick T ₁ , T ₂ , T ₃ are known
	Cooling water Jacket dia =90 mm
	Thermal conductivity of materials $K_1 = K_2 = K_3 = K$ which is known

	Considering the composite slab made up of three materials of thickness L_1 , L_2 , and L_3 having thermal conductivities K_1 , K_2 and K_3
	heat transfer can be calculated using Fourier's Law as
Observation Table	$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$
Calculation	At steady state the rate of heat flow through the individual layers are equal So we can write the following set; $q/A = K_1/L_1 [(T_1+T_2)/2-(T_3+T_4)/2]$ $q/A = K_2/L_2 [(T_3+T_4)/2-(T_5+T_6)/2]$ $q/A = K_3/L_3 [(T_5+T_6)/2-(T_7+T_8)/2]$ $T_1 \& T_2 =$ Interface temperature of 1 st brick and heater, °C $T_3 \& T_4 =$ Interface temperature of 1 st and 2 nd brick, °C $T_5 \& T_6 =$ Interface temperature of 2 nd and 3 rd brick, °C $T_7 \& T_8 =$ Top surface temperature of 3 rd brick, °C $T_7 \& T_8 =$ Top surface temperature of 3 rd brick, °C $T_7 \& t_8 =$ Top surface temperature of 3 rd brick, °C
	system. Check it.
Remarks	• Keep all the assembly undisturbed.
	• Remove air gap between plates by moving hand press gently.
Lah Manual	Operate selector switch of temperature indicator gently.
LaD Manual Experimental	NII Schematic Diagram:
Graphics	Schematic Diagram:
Relevant IS	
Code	0 Define meters filmet to an few
Quiz	 9. Define rate of heat transfer. 10. How will you determine rate of heat transfer of a rectangular, cylindrical slab?

COURSE-<mark>Diploma in Chemical Engineering</mark> SEMESTER-4th

Mechanical Operation Laboratory

EXPERIMENT-1

To Determine Crushing Efficiency of a Roll Crusher

INTRODUCTION:	It is a type of secondary crusher used for further crushing of materials that obtain from primary crushing process.		
SPECIFICATION:	Although not widely used in the minerals industry, roll crushers can be effective in handling friable, sticky, frozen, and less abrasive feeds, such as limestone, coal, chalk, gypsum, phosphate, and soft iron ores. Roll crusher operation is fairly straightforward: the standard spring rolls consist of two horizontal cylinders that revolve toward each other The gap (closest distance between the rolls) is determined by shims which cause the spring-loaded roll to be held back from the fixed roll. Unlike jaw and gyratory crushers, where reduction is progressive by repeated nipping action as the material passes down to the discharge, the crushing process in rolls is one of single pressure.		
	1. Dust / Asthma - Wear approved PPE – dust mask		
	2. Noise - Wear approved PPE – ear muffs or ear plugs		
WARNING:	 3. Electrocution - Ensure system is linked to RCD 4. Entanglement - Ensure all guards are fitted and secure. Use barriers if required to block access 		
	5. Explosion - Ensure fans and ventilation do not allow build up of grain dust		
DESCRIPTION:	Roll crushers are also manufactured with only one rotating cylinder, which revolves toward a fixed plate. Other roll crushers use three, four or six cylinders, although machines with more than two rolls are rare today. In some crushers the diameters and speeds of the rolls may differ. The rolls may be gear driven, but this limits the distance adjustment between the rolls. Modern rolls are driven by V-belts from separate motors. The disadvantage of roll crushers is that, in order for reasonable reduction ratios to be achieved, very large rolls are required in relation to the size of the feed particles. They therefore have the highest capital cost of all crushers for a given throughput and reduction ratio.		
	The action of a roll crusher, compared to the other crushers, is amenable to a level of analysis. Consider a spherical particle of radius <i>r</i> , being crushed by a pair of rolls of radius <i>R</i> , the gap between the rolls being d. If μ is the coefficient of friction between the rolls and the particle, 2θ is the angle formed by the tangents to the roll surfaces at their points of contact with the particle (the angle of nip), and <i>C</i> is the compressive force exerted by the rolls acting from the roll centers through the particle center, then for a particle to be just gripped by the rolls, equating vertically and $\cos\theta = (R+r)/(R+d)$		

Rolling Mill:	
	$F \sin \theta$ R R H $F \mu \cos \theta$ L L
Operating Principles:	To determine the crushing efficiency of roll crusher.
Operating Procedures:	 Switch 'ON' the Main supply. Switch 'ON' the machine followed by MCB. Before charging the feed material, note down the following points o Clean the stuck materials in between two rolls before running the equipment.
	 Properly check the nuts & bolts at the joints. Proper placement of product collector bath. Check the contact points for any wear, & friction.
	4. After completion of the crushing operation, Switch 'OFF' the machine along with MCB connection, then cut the main supply.5. Before leaving, clean the equipment properly.

Observation		 	
Table:	Sl No.		
	1		
	2		
	3		
	4		

Calculation:	
Video Link:	https://www.youtube.com/watch?v=ISJ-WUJFfHs

COURSE-<mark>Diploma in Chemical Engineering</mark> SEMESTER-4th

Mechanical Operation Laboratory

EXPERIMENT-1

To Determine Crushing Efficiency of a Jaw Crusher

INTRODUCTION:	The machine jaw crusher has been designed for reducing the aggregate size. It is used in cement and chemical industries, engineering colleges, govt. departments and other testing laboratories.			
SPECIFICATION:	Laboratory Jaw crusher is designed for fast crushing of aggregates, ores, minerals, coal, coke, chemicals and other similar materials. It is compact and of rugged construction for general laboratory or small pilot plant operations. Two jaws of manganese steel are provided in this laboratory jaw crusher. The moveable jaw produces two blows for every revolution, thus reducing over sizing to a minimum. A combination of forward and downward stroke with a rocking action exerts pressure on the coarser material, yet permits the finished material to pass through the jaws. A hopper is provided at the top for feeding material. The smooth jaws ensure a uniform product.			
WARNING:	Please insert 40 NO. Mobile oil before using the jaw crusher. One cup cover is adding with the jaw crusher. Don't use empty Jaw Crusher.			
	It has one steel channel frame which electric motor and main body of jaw crusher are mounted. The crusher is made of steel. The jaws have forward and downward strokes with sufficient rocking action to throw the crushed material down to the jaws.			
DESCRIPTION:	A hopper is provided at the top for pouring material. Jaw release handle is provided to release the jaw for adjustment adjustable nut is provided to adjust the jaw to get desired size of material. Before operation, the jaw release handle is to be locked in position. It has two bearing grease cups for lubrication. The crusher has one triple v-belt fly wheel- pulley and one wheel.			
	Electric motor is suitable for operation on 440 volts, 7.5 SHP 50 Hz AC supply. Main lead is to be connected to suitable power connection and ensure that the electric motor is properly earthed. Feed the material through hopper.			
	Release the jaw release handle and adjust the jaw with the help of jaw adjustable nut to get desired size of material. Lock the release handle and switch on the mains.			
	To increase or decrease the specimens size of adjust nut in or out after release the handle. Before operation, lock the release handle.			

Parts of Jaw Crusher:	
	 Feed Chute, 2. Drive Flywheel, 3. V-belts, Electric motor, 5. Motor Stand, Settings and Damping Group, 7. Hydraulic Cylinder, Toggle Plate, 9. Swing Jaw, 10. Discharge, Fixed Jaw
Operating Principles:	Through the motor's wheels, the eccentric shaft is driven by the triangle belt to make the movable jaw rotate in a regulated track. Movable jaw plate periodically comes to fixed jaw plate, and away from fixed jaw plate accompany with the movement of movable jaw part. Therefore, the materials in the crushing cavity composed of fixed jaw plate, movable jaw plate and side liners can be crushed and discharged through the discharging opening.
Operating Procedures:	 Switch 'ON' the Main supply. Switch on the machine followed by MCB.
	3. Before charging the feed material, note down the following points
	Properly check the nuts & bolts at the joints. Proper placement of product collector bath. Check the contact points for any wear, & friction.
	4. After completion of the crushing operation, Switch 'OFF' the machine along with MCB connection, then cut the main supply.
	5. Before leaving, clean the equipment properly.

Table:	SI. No.		
	1		
	2		
	3		
	4		

Calculation:	
Video Link:	https://www.youtube.com/watch?v=zvkEn6oytV8&t=51s

COURSE-Diploma in Chemical Engineering

SEMESTER-4th

Mechanical Operation Laboratory

EXPERIMENT- 2

To Study the Screen Analysis & Determine Average Particle Size of Solid Particles in a Sieve Shaker Characteristics in a Sieve Shaker

Introduction	It is used for automatic sieving of finely grind materials in a stacked column of standard sieves from the top (larger openings) to the bottom (smaller opening) according to the particle size.			
	The single of a particle on the size distribution of a mixture of particles can be determine by using several methods. Such as screening gravity, centrifugal sedimentation etc. one of the cheapest and easiest method of particle size analysis is screening. Particle must be separated according to their size before using them for any industrial application. Since, the process efficiency very much dependent and the particle size employed.			
	The most common method being the volume-surface mean diameter.			
	(1)Volume –surface mean diameter:- DVS= 6 $\Phi_S \rho_P A_W = 1/\sum x_i/D_{pi}$ Where Φ_s = sphericity A _W =Total Surface Area ρ_p =density of particle X _i =i th mass fraction D _{pi} =i th average particle diameter			
	(2) Arithmetic mean diameter:- $D_N = (\sum Xpi/D_{pi}^2) / \sum X_{pi} / D_{pi}^3$			
	(3) Mass mean diameter:- $D_w = \sum X_i D_{pi}$			
	(4) Volume mean diameter:- $D_P=1/\int d\phi/D_p$ where, Φ =cumulative mass fraction.			
Theory	The volume mean diameter is calculated by taking the inverse of area under the curve of a graph plotted between Φ vs. $1/D_{\rm pi}$			
Objective	To determine the average particle size of a mixture of particles by Sieve analysis.			

<u>Apparatus</u>	4-200 mesh BSS Sieves, Sieve shaker, brush, sieve opener, balance with weight box
<u>Procedure</u>	Step -1Take 500 gm of sample by coning and quartering method.Step-2Arrange a standard set of sieves serially in a stack the largest mess at the bottom.Step-3Take the sample on the top screen. Shake the whole sieve stack for 10 min in a sieve shaker.Step -4Take the particles left in each screen carefully and separately weight them to find the mess in each screen.Step -5Calculate the mass fraction in each case.Step-6Substitute the formula and find out all the average particle diameters.
<u>Observation</u> <u>Table</u>	Sl.MeMeAvMaMaMa $1/D$ Xi/Xi/Xi/Xi/Xi/DpiNossssg.sssssspipi D_{pi} D_{pi}^2 D_{pi}^3 D_{pi} noopepartcollfracicleectetionpi D_{pi} D_{pi}^2 D_{pi}^3 D_{pi} g(gm2)>Image: Description of the second secon
<u>Calculation</u>	
<u>Precautions</u>	 Ro-Tap Sieve Shaker: A Ro-Tap sieve shaker having provision to hold a stack of 10 nos. of 200mm dia. standard sieves, 1 no. receiver pan and top lid. Capacity: The unit should be run with a motor of suitable capacity, running on 230V/1Ph/50Hz electric supply.
Lab Manual	r unning on 200 v/ 11 n/ Son2 ciccu ic supply.
<u>Experimental</u> <u>Graphics</u>	https://www.youtube.com/watch?v=NltqDoq7dMw

COURSE-Diploma in Chemical Engineering

SEMESTER-4th

Mechanical OperationLaboratory

EXPERIMENT-3

To Study of Grinding Characteristics of Ball Mill & its Critical Speed
Introduction	A ball mill is a type of grinder used to grind materials into extremely fine powder for use in mineral dressing processes, paints, pyrotechnics, and ceramics.
Theory	A ball mill is a horizontal cylinder partly filled with steel balls (or occasionally other shapes) that rotates on its axis, imparting a tumbling and cascading action to the balls. Material fed through the mill is crushed by impact and ground by attrition between the balls. The grinding media are usually made of high-chromium steel. The smaller grades are occasionally cylindrical rather than spherical. There exists a speed of rotation (the "critical speed") at which the contents of the mill would simply ride over the roof of the mill due to centrifugal action. The mill is usually divided into at least two chambers,(Depends upon feed input size presently mill installed with Roller Press are mostly single chambered), allowing the use of different sizes of grinding media. Large balls are used at the inlet, to crush clinker nodules (which can be over 25 mm in diameter). Ball diameter here is in the range 60– 80 mm. In a two-chamber mill, the media in the second chamber are typically in the range 15–40 mm, although media down to 5 mm are sometimes encountered. As a general rule, the size of media has to match the size of material being ground: large media can't produce the ultra-fine particles required in the finished cement, but small media can't break large clinker particles. A current of air is passed through the mill. This helps keep the mill cool, and sweeps out evaporated moisture which would otherwise cause hydration and disrupt material flow. The dusty exhaust air is cleaned, usually with bag filters.
	Feed Product Product
Objective	To study the working principle & critical speed of Ball mill.

<u>Critical Speed</u>	The formula for critical speed is $CS = \frac{1}{2}\pi \sqrt{(g/(R-r))}$ where g is the gravitational constant, R is the inside diameter of the mill and r is the diameter of one piece of media.					
<u>Procedure</u>	 Switch 'ON' the Main supply. Switch 'ON' the machine followed by MCB. Before charging the feed material, note down the following points. Clean the inner side of the cylindrical shell properly. Set the RPM of the shell below the critical speed of the mill. Properly check the nuts & bolts at the joints. Proper placement of product collector bath. Check the contact points for any wear, & friction. After completion of the grinding operation, Switch 'OFF' the machine along with MCB connection, then cut the main supply. Before leaving, clean the equipment properly. 					
<u>Observation</u> <u>Table</u>	SI No.					
<u>Calculation</u> <u>Precautions</u>	 Clean the mixer before and after operating. Maintain enough distance form mixer while operating. Always remove power supply before pouring the contents out from mixer 					
<u>Experimental</u> <u>Graphics</u>	https://www.youtube.com/watch?v=aVQ9B3LtCPk					

COURSE-Diploma in Chemical Engineering

SEMESTER-4th

Mechanical OperationLaboratory

EXPERIMENT-4

To Study the Filtration Characteristics of a Slurry in a Filter Press

An industrial filter press is a tool used in separation processes, specifically to separate solids and liquids. The machine stacks many filter elements and allows the filter to be easily opened to remove the filtered solids, and allows easy cleaning or replacement of the filter media.
Filter presses cannot be operated in a continuous process but can offer very high performance, particularly when low residual liquid in the solid is desired. Among other uses, filter presses are utilised in marble factories in order to separate water from mud in order to reuse the water during the marble cutting process
Filter is the process whereby a solid is separate from a liquid or gas by means of porous medium which retain the solid but allows the fluid to pass. A filter cake gradually builds upon the medium and the resistance to flow problem can be regarded as two parts: 1. Through filter medium.
2. Through bed formed by the particles on the medium
3. The plates and frames arranged alternatively are supported on a pair of rails. Plate has a ribbed surface and press is closed by means of a screw. A chamber is therefore formed between each pair of successive frames.
To determine the characteristic of plate and frame filter press at a constant pressure and to calculate the filter cake resistance 'a' and

<u>Apparatus</u>	Plate and fr	ame filter p	oress, Calcium	Carbonate sl	urry, stop clock.
<u>Procedure</u>	The plate a medium be tank by clos stirrer and the vessel. collected in is followed This slurry The slurry to 15psi us The pure f seeing the noted for e of filtration filter press taken off th dried in ai noted.	nd frame fi tween the fi sing the dis the pressu The outlet the collect 5 liters of is poured i is kept agita ing the valv ilter water level in the very 1 cm. a, valves ar is carefull e medium a r for 24 hr	lter press is ar frames. Few lit charge value. T re is adjusted valve is opene ion tank. After 3% calcium c nto the tank by ated by stirrer ve in the vesse is collected i level glass a s rise in the leve re closed and y opened. A su and is weighed rs. After that	ranged by ke ers of water The water is to 15 psi usi d. The pure that the follo arbonate slu v closing the and the pres l. The outlet n the collec stop clock is el glass. After system is sy mall quantity l using weigh	eeping wet filter is pour in to the kept agitated by ng the valves in filtered water is owing procedure rry is prepared discharge valve ssure is adjusted valve is opened tion tank. After started. Time is the completion vitched off. The y of wet cake is aing balance and e weight is also
<u>Observation</u> <u>Table</u>	Height h (cm)	Total time, t (sec)	Volume of filtrate collected V= A _c x h(m ³)	t/V (sec/m³)	Height h (cm)
<u>Calculation</u>	1. 1. Con 1. 2. To experimenta	Calculate I data is us	of the feed slur $C = \frac{Cs}{1 - [(mw/md) - 1]}$ the cake ar ed and a graph Slope Intercept	Try, C $1]Cs/\rho$ and medium is plotted as	resistances th

<u>Precautions</u>	 Size of the filter press, L =200mm, B =200mm No. of plate = 7 No. of frames = 6 No. of chambers formed, n = 12 Area offered by one chamber, A = L x B = 40000 mm² Total filter area, A_f = n x A = 12 x 40000 = 480000mm² Pressure drop p (observed from pressure gauge) = Kg/m² p = 1.0 - 0.85 = .15 Kg/m² Diameter of the collection tank, D_c = 200mm Area of the collection tank, A_c = 31400mm²
I ah Manual	 ➢ Wet cake, m_w = 420gms ➢ Dry cake, m_d = 400 gm ➢ Slurry concentration, C_s = 5%
<u>Experimental</u> <u>Graphics</u>	https://www.youtube.com/watch?v=j8hR9cP87tQ
<u>Relevant IS</u> <u>Code</u>	
Quiz	

Mechanical Operation Laboratory

EXPERIMENT-5

To Study the Solid-Liquid separation characteristics in a centrifuge.

Introduction	Solid-liquid separation through centrifugation is often a crucial step to obtain the desired product after precipitation or crystallization. There are two types of centrifugation (also called dewatering or deliquoring): filtering and sedimenting. This guide focuses mainly on filtering centrifugation because it is the more common type of solid- liquid separation in chemical and pharmaceutical production applications.
	It consist of a rotating bucket mode of stainless steel with performs on side wall which is roted by a motor to a friction clutch. The designing of the friction clutch is such that the minimum spirit of the bucket is gain gradually. The centrifuge holes a mass of materials and by the application of the centrifugal force removes liquids from the solid through the process.
Theory	Solid-Liquid Separation (with overflow by gravity) Fest-Flüssig-Trennung (mit freiem Überlauf) Image: Solid Sector S
Objective	To study the solid-liquid separation characteristics of a solid-liquid mixture by using centrifuge.

<u>Apparatus</u>	Compro Chamb Collect Agents	Compressor, Electricity/Battery, Control panels, Floatation Chamber, SS/MS structure for hold up the equipment, Froth Collecting Tank, Agitator, Diffuser Material for Feed, Frothing Agents and Collectors & drainage system.						
<u>Procedure</u>	A certa mixed top cov dischar the equ measur taken, i at least	A certain quantity of liquid and a certain amount of solid are mixed in a bucket and are charged to the centrifuge. Close the top cover of the centrifuge and start the equipment. After the discharge of liquid through the outlet of the centrifuge. Mark the equipment starting & ending time. The liquid volume was measured similarly. In the next run, another quantity of solid is taken, keeping the same liquid volume. Repeat the experiment at least three times with the different quantity of solid & liquid.						
<u>Observation</u> <u>Table</u>	SI No. 1 2 3	Weight of Solid (g)	Volume of Liquid (ml)	Collection time of Filtrated (sec)	Volume of filtrated Collected (ml)			
<u>Calculation</u>	The r	ate of filtra	ation = volun collec	ne of filtrated co cting time.	ollected/Filtrated			
Lab Manual								
<u>Experimental</u> <u>Graphics</u>	<u>https:/</u>	<u>/www.you</u>	<u>itube.com/w</u>	<u>vatch?v=laZdnM</u>	<u>5hFtc</u>			
Quiz								

COURSE-Diploma in Chemical Engineering

SEMESTER-4th

Mechanical Operation Laboratory

EXPERIMENT-6

To Study the Solid-Liquid Mixing Characteristics in a Sigma Mixer

Introduction	The sigma blade mixer is a commonly used mixer for high viscosity materials. The Sigma Mixer with extruder is used for mixing, kneading and extruding of highly viscous mass, sticky and dough like products, Mixing of pastes, rubber, and heavy plastic masses, applications in Food, Rubber, Pharmaceutical, Chemical and agro industries, Dry powder to wet phase mixing.
Theory	The universal mixing and kneading machine consists of two counter-rotating blades in a rectangular trough curved at bottom to form two longitudinal half cylinders and a saddle section. The blades are driven by gearing at either or both ends. The mixing action is a combination of bulk movement, smearing, stretching, folding, dividing and recombining as the material is pulled and squeezed against blades, saddle and sidewalls. The blades are pitched to achieve end-to-end circulation. Rotation is usually such that material is drawn down over the saddle. The blades run at different speeds, with the advantages of faster mixing from constant change of relative position. The blade design most widely used is the sigma blade. The sigma blade mixer is capable of starting and operating with either liquids or solids or a combination of both. The sigma blade has good mixing action, readily discharges materials and is relatively easy to clean when sticky material are being processed. The drive is given through an electric motor coupled to a reduction gearbox. The whole assembly is fitted on rigid MS structure. The tilting mechanism is provided for discharge of product.
Objective	To study the working principle of Sigma mixer.

<u>Apparatus</u>	Apparatu Double U Stainless Drive: 7.5 arrangem even in 1 switch for	s consist of shaped Capa Steel, 2 Nos. 1 5 HP motor ent is provid running cond r mains and M	Mixing Troug acity 7 kg (app moving in oppo coupled to Re- ed so as to tilt dition. Control fotor, Mains In-	gh Material rox.) Sigma site directio duction Gea the contain Panel com dicator.	Stainless Steel, Blades: Material on with AC Motor ar Box. A Tilting er from (0-110°) prises of on off
<u>Procedure</u>	 Ensu Now Cont Switt Switt Switt Mix Switt Remt Remt Tilt Cleat 	are all switch feed the mat ach on mains of ach on Motor the feed as de ach off motor tove power su the mixer and n the mixing	es are in off pos terial to be mixe upply to machin on/off esired upply d collect the mixe area with wate	sition ed in mixer ne ĸ. r.	
<u>Observation</u> <u>Table</u>	 Wei Wei Wei Wei St No. 1 2 3 4 5 	ight of the cruc ight of the cruc ight of the cruc <i>Weight of</i> <i>mixture (g)</i>	cible 1=g cible 2 = g cible 3= g <i>Weight of Dry</i> <i>Mixture (g)</i>	Weight of Water (g)	Consistence of Water (%)
<u>Calculation</u>					

<u>Precautions</u>	 Clean the mixer before and after operating. Maintain enough distance form mixer while operating. Always remove power supply before pouring the contents out from mixer.
<u>Lab Manual</u>	
<u>Experimental</u> <u>Graphics</u>	https://www.youtube.com/watch?v=1fWI1MIu80k
<u>Relevant IS</u> <u>Code</u>	
<u>Quiz</u>	

- -

COURSE-Diploma in Chemical Engineering

SEMESTER-4th

Mechanical OperationLaboratory

EXPERIMENT- 7

To determine the Screening characteristics in a Vibratory Screen

Introduction	It is a device made with a screening surface vibrated mechanically at high speeds and used especially for screening ore, coal, or other fine dry materials. Vibrating screens are the most important screening machines primarily utilised in the mineral processing industry. They are used to separate feeds containing solid and crushed ores down to approximately 200 µm in size and are applicable to both perfectly wetted and dried feed
Theory	Let a screening operation is being carried out for two materials A & B. F=mass flow rates of feeds, B= mass flow rates of underflow, A=mass flow rates of overflow. The mass fraction of B in feed, overflow and underflow are $(1-X_F)$, $(1-X_D)$ and $(1-X_B)$. From total mass balance: $F = B + D$
Objective:	To study and operate of the vibrating screen and to find the effectiveness of the screen.

APPARATUS:	Vibra opene	Vibrating screen, BSS Mesh sieves, Sieve shaker, Brush, Sieve opener, Balance, Weight box.						
Procedure	STEP-1: Take 1 kg of sample and sieve it in the sieve-shaker. Write down the mass collected in each screen through 4 to 60 meshes and the cumulative mass fraction.							
	STEP-	2: Mix all	the particl	es and fee	d to the	vibrati	ng screen.	
	STEP- throug	3: Start t gh differe	he motor nt bags afte	and collee er 15 minu	ct the o ites of o	overflov	w and underflo m.	
	STEP- cumul under STEP- the gr at the	4: Sieve ative mas size and c 5: Calcula aph. Ther lower scr	the unit o is fraction to oversize) of the the X_F , in calculate een size -1	versize ar for all. Plot n the same X_B and X_D the effect .5mm= (0.	nd unde t Φ vs. I e graph. at the iveness .0625 ir	ersize a D _P (for f cut poi of the nch).	and calculate th feed, oversize an nt diameter fro screen (E=E _A *E	
	Sl No.	Mass Flow rate (F)	Underflow rate (B)	Overflow rate (D)	EB		Effectiveness (E)	
	1							
	2							
<u>Observation</u> Table	3							
	4							
	5							
<u>Calculation</u>								
Lab Manual								
<u>Experimental</u> Graphics	<u>https:</u> <u>https:</u>	//www.yo //www.yo	utube.com/ utube.com/	<u>/watch?v=s</u> /watch?v=b	dWzgng B5k4eti	<u>gGLQ</u> nsLY		
arapines								

Mechanical Operation Laboratory

EXPERIMENT-8

To Study the Filtration characteristics in a Vacuum Filtration Apparatus with Buckner Funnel



<u>Apparatus</u>	Buchner funnel vacuum filtration setup requires the following apparatus: filter flask, filter trap, water pump, a gadget that makes the system efficient into the aspirators-which works on the principle of blocking access into the vacuum when water is passing over as is seen in the water tap system. Other equipment used in assembling the Buchner funnel vacuum filtration apparatus is; the non-reusable filter funnel and a filtrate receptacle.							
<u>Procedure</u>	Fit the n mounting t by using a d The side- a thick rub vacuum tra Place a Erlenmeyer Buchner fu	Fit the non-reusable filter funnel into the filtrate receptacle. After mounting the funnel and the filtrate on the base, a vacuum is applied by using a connection passage linking to the receptacle. The side-arm of the Erlenmeyer flask is clamped to a ring stand and a thick rubber hose is attached to it. The tubing is now connected to a vacuum trap as well as to the aspirator. Place a rubber sleeve and Buchner funnel on top the sidearm Erlenmeyer flask. Place a filter paper that fits squarely into the Buchner funnel.						
<u>Observation</u> <u>Table</u>	<i>St No.</i> <u>1</u> 2 3 4 5							
<u>Calculation</u>			(
<u>Experimental</u> <u>Graphics</u>	https://ww	https://www.youtube.com/watch?v=fFdvEgg1t14 https://www.youtube.com/watch?v=U3a8mzJIKq8						
Quiz								

Mechanical Operation Laboratory

EXPERIMENT-9

Demonstration of Centrifugal & Gear Pump

DEFINATION:	Pump is a mechanical device using suction or pressure to raise or move liquids, compress gases or force air, sometimes slurries into inflatable objects.					
SPECIFICATION:	 Pump are classified in two types: (i) Centrifugal pump & Positive displacement pump (ex: gear pump) A centrifugal pump consists of a rotating shaft that is connect an impeller, which is usually comprised of curved blades impeller rotates within its casing and sucks the fluid through the of the casing. The fluid's kinetic energy increases due to the eradded by the impeller and enters the discharge end of the cather that has an expanding area. The pressure within the fluid increaccordingly. A gear pump is a type of positive displacement (PD) pump. pumps use the actions of rotating cogs or gears to transfer fl The rotating gears develop a liquid seal with the pump casing create suction at the pump inlet. Fluid, drawn into the pum enclosed within the teeth of the rotating gears and transferr the discharge. A gear pump delivers a smooth pulse-free proportional to the rotational speed of its gears. 					
THEORY:	1. GENERAL PUMP THEORY Consider the pump shown in Figure. The work done by the pump, per unit mass of fluid, will result in increases in the pressure head, velocity head, and potential head of the fluid between points 1 and 2. Therefore: work done by pump per unit mass = W/M increase in pressure head per unit mass = (P ₂ -P ₁)/ ρ increase in velocity head per unit mass = (V ₂ ² -V ₁ ²)/2 increase in potential head per unit mass = g(z ₂ -z ₁) in which: <i>W</i> : work done <i>M</i> : mass <i>P</i> : pressure ρ : density <i>v</i> : flow velocity <i>g</i> : acceleration due to gravity <i>z</i> : elevation Applying Bernoulli's equation between points 1 and 2 in Figure 1 results in: $\frac{W}{Mg} = (P_2-P_1)/\rho g + (v_2^2-v_1^2)/2g + (z_2-z_1)$					









Mechanical OperationLaboratory

EXPERIMENT-10

To Study the Solid-Solid Separation in a Froth-Floatation Cell.

Introduction	Froth flotation is a process for selectively separating hydrophobic materials from hydrophilic. This is used in mineral processing, paper recycling, and waste-water treatment industries.
Theory	Froth flotation involves separation of a mixture on the basis of difference in the surface properties when the mixture is suspended in the aerated liquid, the gas bubbles (foam) adhere to one of the constituents- the one which is more difficult to wet by the liquid, where as the other constituents being wet stay with the liquid. Froth flotation is employed widely in metallurgical industries (sulphide ores) and coal. Mixed liberated particles can be separated from each other by flotation if there are sufficient differences in their wet ability. The flotation process operates by preparing a water suspension of a mixture of relatively fine-sized particles (smaller than 150 micrometers) and by contacting the suspension with a swarm of air bubbles in a suitably designed process vessel. Particles that are readily wetted by water (hydrophilic) tend to remain in suspension, and those particles not wetted by water (hydrophobic) tend to be attached to air bubbles, levitate (float) to the top of the process vessel, and collect in a froth layer. Thus, Differences in the surface chemical properties of the solids are the basis for separation by flotation. Surfaces that do not have strong surface chemical bonds that were broken tend to be non-polar and are not readily wetted. Substances such as graphite and talc are examples that can be broken along weakly bonded layer planes without rupturing strong chemical bonds. These solids are naturally flotable. Also, polymeric particles possess non-polar surfaces and are naturally hydrophobic. By contrast, most naturally occurring materials are polar and exhibit high free energy at the polar surface. The polar surfaces react strongly with water and render those particles naturally hydrophobic. By contrast, most naturally occurring materials are polar and exhibit high free energy at the polar surface chemical agents that are adsorbed selectively on the particle surface.
objective	(ex: coal and sand) and to find the separation efficiency.

<u>Apparatus</u>	Compressor, Electricity/Battery, Control panels, Floatation Chamber, SS/MS structure for hold up the equipment, Froth Collecting Tank, Agitator, Diffuser Material for Feed, Frothing Agents and Collectors & drainage system.					
<u>Procedure</u>	 Accurately weigh 45 gm of finely ground coal and 5 gm of sand. Mix them thoroughly. Add 10 Litters of water to the cell and start the impeller. Add 2/3 drops of pine oil (floating agent and collector) and a pinch of detergent to create a stable froth. Add the coal mixture to the tank. Skim off the froth and collect the same through the weir. Dry the froth collected and find the separation efficiency. 					
<u>Observation</u> <u>Table</u>	Sl No. 1 2 3 4 5					
<u>Calculation</u>	Wt. Of initial coal taken: W1 = Wt of sand taken: W2 = Wt of dried froth: W3 = Separation efficiency: (W3/W1) x 100 =%					
<u>Experimental</u> <u>Setup</u>	The unit consists of a froth flotation cell made SS 304 steel with an impeller with a diffusion ring. An air inlet is provided at the top. The same is connected to a ¼ hp motor through reduction pulley system. A weir attached to the cell helps in collection of the froth.					
Lab Manual						
<u>Experimental</u> <u>Graphics</u>	https://ww	w.youtube	.com/watch?v	=E2Ln8Kgrh	ıpA	
<u>Relevant IS</u> <u>Code</u>						
Quiz						

Mechanical Operation Laboratory

EXPERIMENT- 11

To Study the Fluidisation Characteristics of Sand in a Fluidisation Apparatus

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Introduction	Fluidized sand beds (FSBs) have played an important role in the evolution of recirculation aquaculture systems (RAS), but technical problems have limited their application. Recent advances in FSB design have improved hydraulic efficiency, reliability, installation costs and maintenance.
	A fluidized bed is a physical phenomenon occurring when a quantity of a solid particulate substance (usually present in a holding vessel) is placed under appropriate conditions to cause a solid/fluid mixture to behave as a fluid. This is usually achieved by the introduction of pressurized fluid through the particulate medium. This results in the medium then having many properties and characteristics of normal fluids, such as the ability to free flow under gravity, or to be pumped using fluid type technologies.
	The resulting phenomenon is called fluidization. Fluidized beds are used for several purposes, such as fluidized bed reactors (types of chemical reactors), solids separation, fluid catalytic cracking etc.
	A fluidised bed consists of fluid-solid mixture that exhibits fluid-like properties. As such, the upper surface of the bed is relatively horizontal, which is analogous to hydrostatic behavior. The bed can be considered to be a heterogeneous mixture of fluid and solid that can be represented by a single bulk density.
Theory	Furthermore, an object with a higher density than the bed will sink, whereas an object with a lower density than the bed will float, thus the bed can be considered to exhibit the fluid behavior expected of Archimedes' principle. As the "density", (actually the solid volume fraction of the suspension), of the bed can be altered by changing the fluid fraction, objects with different densities comparative to the bed can, by altering either the fluid or solid fraction, be caused to sink or float.
	In fluidised beds, the contact of the solid particles with the fluidisation medium (a gas or a liquid) is greatly enhanced when compared to packed beds. This behavior in fluidised combustion beds enables good thermal transport inside the system and good heat transfer between the bed and its container. Similarly, to the good heat transfer, which enables thermal uniformity analogous to that of a well-mixed gas, the bed can have a significant heat-capacity whilst maintaining a homogeneous temperature field.



<u>Apparatus</u>	Experiments were carried out in Plexiglas equipment with an inner diameter of 300 mm and a height of 8250 mm, as shown in Fig. 2. A plate distributor with 100 holes of diameter 3 mm was fixed in the bottom of the fluidized bed. The opening area ratio is 1.1 %					
<u>Procedure</u>	The pressures at different positions along the bed height were measured by using a pressure transducer and the air superficial velocity was measured by a rotameter. The initial and dense bed height was measured by using a ruler adhered on the wall of the bed. There were many measuring points on the wall along the bed height. More measuring points were installed in the dense bed. The average solid holdup ε_s can be calculated by the following two equations, $\Delta P = \Delta H \times g \times ((1 - \varepsilon_s)\rho g + \varepsilon_s\rho_p) \approx \Delta H \times g \times \varepsilon_s\rho_p \Delta P = \Delta H \times g \times ((1 - \varepsilon_s)\rho g + \varepsilon_s\rho_p) \approx \Delta H \times g \times \varepsilon_s\rho_p \Delta H \times g \times \varepsilon_s\rho_p$ (1) $\varepsilon_s = \Delta P \Delta H \times g \times \rho_p \varepsilon_s = \Delta P \Delta H \times g \times \rho_p$ (2) where ΔP means the pressure drop, kPa; ΔH is the distance between two measure points, m; ρ_p is the density of particles, kg/m ³ .					
	Sl No.					
	1				_	
	2					
<u>Observation</u> <u>Table</u>	3					
	4					
	5					
		•	•	·		
<u>Calculation</u>						
<u>Calculation</u> <u>Experimental</u> <u>Graphics</u>	https://www.yo	outube.com/wat	<u>ch?v=xE0dyQ</u>	<u>SRTB4</u>		

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	Drying: The term drying refers generally to the removal of moisture from a substance. It is one of the oldest, most commonly used and most energy consuming unit operation in the process industries					
	Drying Rate: Drying rate is the amount of moisture removed per unit time. N					
	Affecting factors:					
	1. Flow rate of air					
Introduction	2. Solid characteristics					
	3. Tray material.					
	Laboratory methods:					
	Drying process of a material can be described as a series of steps in which drying rate plays a key role. Following figure shows typical drying rate curve for a constant drying conditions. Point B represents equilibrium temperature conditions of the product surface. Section B to C of the curve, known as the constant rate periods, represents removal of unbound water from the product. The water acts as if the solid is not present. The surface of the product is very wet and water. And the water activity is equal to one. The constant rate period continues as long as the amount of water evaporates is equal to the amount of water supplied to the surface of the material.					
Theory	FREE MOISTURE (kg H20 / kg dry solid)					
	The falling rate period is reached when the drying rate starts to decrease, and the					
	internal flow of liquid or vapor. This point is represented by C in the figure. At this					
	point there is not enough water on the surface to maintain a water activity value of one. The falling rate period can be divided into two steps. A first falling drying rate					
	occurs when wetted spots in the surface continually diminish until the surface is dried					
	(Point D). Second falling rate period begins at point D when the surface is completely dry. The					
	plane of evaporation recedes from the surface. Heat required for moisture removal is transferred through the solid to the vaporization of moisture in the solid and the vapor					



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Procedure	 Measure the weight of the dry solid (W_s) Add water Measure the weight of the wet solid. This will give initial moisture content. Put the wet solid inside the dryer and spread evenly. Start the dryer At different time interval measure the weight of the sample from the attached balance. Difference between two successive weights will give the moisture content. Plot time vs moisture content curve Measure the area of the tray (a). Calculate the drying rate for each step. Plot moisture content vs drying rate. 						
Observation Table	Sl No. 1 2 3 4 5	<i>Time(t)</i>	Δt	Moisture content (X)		Drying Rate (N)	
Calculation		Calcula	te drying ra the form J = -(W _s ΔΣ	ate (N) using ula X)∕a∆t			
Video	https://www.youtube.com/watch?v=cjIekv_IW60						
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	Drying: The term drying refers generally to the removal of moisture from a substance. It is one of the oldest, most commonly used and most energy consuming unit operation in the process industries					
	Drying Rate: Drying rate is the amount of moisture removed per unit time. N					
	Affecting factors:					
	1. Flow rate of air					
Introduction	2. Solid characteristics					
	3. Tray material.					
	Laboratory methods:					
	Drying process of a material can be described as a series of steps in which drying rate plays a key role. Following figure shows typical drying rate curve for a constant drying conditions. Point B represents equilibrium temperature conditions of the product surface. Section B to C of the curve, known as the constant rate periods, represents removal of unbound water from the product. The water acts as if the solid is not present. The surface of the product is very wet and water. And the water activity is equal to one. The constant rate period continues as long as the amount of water evaporates is equal to the amount of water supplied to the surface of the material.					
Theory	$w_c = critical moisture content$ First falling rate period EREE MOISTURE (kg H ₂ 0 / kg dry solid)					
	The falling rate period is reached when the drying rate starts to decrease, and the					
	surface water activity falls to less than one. The rate of drying is governed by the internal flow of liquid or vapor. This point is represented by C in the figure. At this					
	point there is not enough water on the surface to maintain a water activity value of					
	occurs when wetted spots in the surface continually diminish until the surface is dried					
	(Point D). Second falling rate period begins at point D when the surface is completely dry. The					
	plane of evaporation recedes from the surface. Heat required for moisture removal is					
	transferred through the solid to the vaporization of moisture in the solid and the vapor					



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Procedure	 Measure the weight of the dry solid Add water Measure the weight of the wet solid. This will give initial moisture content. Put the wet solid inside the dryer and spread evenly. Start the dryer At different time interval measure the weight of the sample from the attached balance. Difference between two successive weights will give the moisture content. Plot time vs moisture content curve Measure the area of the tray. Calculate the drying rate for each step. 						
Observation Table	Sl No. 1 2 3 4 5	<i>Time(t)</i>	Δt	Moisture content (X)		Drying Rate (N)	
Calculation	Calculate drying rate (N) using the formula $N = -(W_s \Delta X)/a\Delta t$						
viaeo	nups:/	/www.you	ude.com/v	vaicn:v=HqP45	sisipw		

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EXPERIMENT-3



	Crystalization: Crystallization or crystallisation is the process by which a solid forms, where the atoms or molecules are highly organized into a structure known as a crystal
	Affecting factors:
	1. Nature of Solvent
	2. Nature of impurities
Introduction	3. Crystalization time
	Laboratory methods:
Theory	Crystallization is a technique for purifying solids that contain small amounts of impurities. This technique is based on the fact that both the solid and the impurities may dissolve in a given solvent, but not to the same extent. Solubility is a function of concentration. If we keep the amount of solvent constant and gradually increase the amounts of solute, eventually we approach a limit beyond which the solute can no longer dissolve because it's too much for the amount of solvent available. Another factor important in solubility is temperature. The amount of solvent available. Another factor important in solubility is temperature. The amount of solvent available. Another factor important in solubility is temperature. The amount of solvent available is solves like" principle. This makes direct reference to polarity. More polar substances dissolve better in polar solvents (for example alcohol in water), whereas less polar substances dissolve better in less polar solvents (for example alcohol in water), whereas less polar substances dissolve better in the solution of salt in water, we can cause some of the salt to come out of solution by evaporating some of the water (changing the concentration), lowering the temperature of the water, or adding a less polar solvent such as rubbing alcohol (decreasing the polarity). In the technique of crystallization, the most commonly manipulated parameter is temperature. First we dissolve the impure solid in a solvent that dissolves both, solid and impurities. We try to dissolve the maximum amount of solude not no room temperature. The amount of solute that the solvent can dissolve at low temperature is lower than at high temperature. Since the concentration of the main solution here solution because of the solid will come out of solution because of the solid on the solid and impurities. We try to dissolve the maximum amount of solute the main solution because of the solid will come out of solution, hopefully in the form of pure crystalls. The impurities, being present in s

Objective	To stud	y crystallizatio	on charecte	eristics		
Procedure	 Measure the weight of the impure solid and its melting point range. Add appropriate amount of heated solvent and dissolve the impure solid. Filter out insoluble impurities. Cool down till crystal starts appearing. Note the time. Cool till room temperature. Filter the mixture to separate mother liquor and crystals. Plot time vs moisture content curve Dry the crystals and determine their weight. Find the difference between weight of impure material and the weight of the crystals. Determine the melting point of the crystal. Compare it with the melting point of the sample with impurities 					
Observation Table	Sample Impure Crystal	Physical Appearance	Melting Point	Weight W1 W2	Time of first appearance of crystal	Temperature at which crystal appears
Calculation	W_1 - $W_2 = \Delta W$ This will give the amount of solute which cannot be recovered.					
Video	https:// <u>w</u>	ww.youtube.co	m <u>/</u> watch?v	=OtopBR	Rpqa4	

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EXPERIMENT-4



	Drying: The term drying refers generally to the removal of moisture from a substance. It is one of the oldest, most commonly used and most energy consuming unit operation in the process industries					
	Drying Rate: Drying rate is the amount of moisture removed per unit time. N					
	Affecting factors:					
	1. Flow rate of air					
Introduction	2. Solid characteristics					
	3. Tray material.					
	Laboratory methods:					
	Drying process of a material can be described as a series of steps in which drying					
	rate plays a key role. Following figure shows typical drying rate curve for a constant drying conditions. Point B represents equilibrium temperature conditions of the					
	product surface. Section B to C of the curve, known as the constant rate periods,					
	represents removal of unbound water from the product. The water acts as if the solid					
	activity is equal to one. The constant rate period continues as long as the amount of					
	water evaporates is equal to the amount of water supplied to the surface of the					
	material.					
	falling constant					
	rate rate					
	$\begin{bmatrix} w_c = critical \end{bmatrix}$					
	S content					
	<u> </u>					
Theory						
I neor y	😇 🛛 🔁 First falling rate period					
	N X					
	HO H					
	Second falling rate period					
	FREE MOISTURE (kg H ₂ 0 / kg dry solid)					
	The falling rate period is reached when the drying rate starts to decrease, and the					
	surface water activity falls to less than one. The rate of drying is governed by the internal flow of liquid or vanor. This point is represented by C in the figure. At this					
	point there is not enough water on the surface to maintain a water activity value of					
	one. The falling rate period can be divided into two steps. A first falling drying rate					
	occurs when wetted spots in the surface continually diminish until the surface is dried (Point D)					
	Second falling rate period begins at point D when the surface is completely dry. The					
	plane of evaporation recedes from the surface. Heat required for moisture removal is					
	transferred through the solid to the vaporization of moisture in the solid and the vapor					

moves through the solid into air stream. The amount water removed in this period can be relatively small compared to the constant rate and first falling rate period. However this period may take much longer than constant rate period because the drying is slow.

Drying rate experiments can be simply done by measuring the weight change during drying. The material to be dried is placed on the tray. The tray is suspended from a balance and exposed to air flow in drying tunnel.



Spray drying is the transformation of feed from a liquid form into dried form by spraying feed into hot drying medium. The feed can either be a solution, suspension or paste. The resulting product conforms to powders, granules or agglomerates. If the liquid is a flammable solvent like ethanol then hot nitrogen is used instead of air. This method is also used of drying of many heat sensitive materials such as foods and pharmaceuticals. Because, some ion can be oxidized in presence of air and that will be harmful for our health. The atomizer is the main thing in spray drying. The atomizer increases the surface area of the liquid solution by creating fine droplets. The mist is sprayed into a chamber of air heated to temperature above the vaporization temperature of the solution's solvent. The contact between mist and hot air makes the vaporization of water. The flow of liquid and gas may be co-current, counter-current or the combination of both (mixed current). In this experiment, mixed current spray dryer was used. The liquid drying rate depends upon the solution flow rate, flow rate of the air, size of the droplets, temperature of the air and solution and amount of solvent in the solution. Cyclone separator is used to separate dried particles from vaporized solvent. The air rotates helically in the cyclone separator. The dried particle is forced to go to the bottom and the air is expelled to the atmosphere pressure goes to a scrubber. The dried product can be in the form of powders, granules or agglomerates depending upon the physical and chemical properties of the feed, the drver design and desired product properties.

Any spray drying system has its major parts like feed delivery and atomization system, a drying chamber, a hot gas production and delivery system, a solid gas separation system and a product discharge system. There are some main parts of a common spray dryer. They are air blower, heater, drying chamber, atomizer, compressor and cyclone separator system. There are three types of atomizer. Two fluid nozzles, pressure nozzle atomizer and centrifugal atomizer. In this experiment, two fluid nozzles atomizer was used. Spray is created by contacting two fluids, the feed and a compressed gas. The atomization energy is provided by the compressed gas, usually air. The contact can be internal or external. It is used in low production rate drying. Because, it ensures the quality of particles size. Two fluid nozzles (Internal contact and external contact) There are mainly four steps involved in the spray drying.

1. Atomization of a liquid feed into fine droplets - Atomization is the process of breaking up the bulk liquid into millions of individual droplets forming a spray. For atomization process, pressure energy is needed. In this experiment, two fluid nozzles were used. The liquid feed entered into the atomizer. Then the liquid stream comes in contact with compressed air. Thus, fine droplets were created. The mist was sprayed into the dryer chamber from the spray

 Normal and the stream of the stream
$\begin{array}{c} \text{Air heating system} \\ \hline \\ Feed tank \\ \hline \\ Feed pump \\ \hline \\ Feed pump \\ \hline \\ Feed pump \\ \hline \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ $

Objective	To study operation of a spray dryer

Procedure	 Some milk powder was taken in a big beaker and the weight of milk powder was taken by weighing. The amount of milk powder was 50 gm. Water was added to prepare a 5% (weight) milk solution. The powder was dissolved thoroughly and was strained thorough a fine cloth. The spray dryer was started by turning the hot air blown on. The heater was adjusted and noted the temperature of hot air. The dryer was allowed running for 10-15 minutes The dryer was run with prepared milk solution for a fixed atomized pressure. At the end, the dryer was run with distilled water for 10-15 minutes 					
Observation Table	Obs. No. 1 2 3 4 5	<u>Re</u> covered milk (M ₁)	Time (t)	Percentage of Milk Recovered	Drying rate	
	Dryi Rec	ng rate = Wei covered milk/	ght of Time			
Calculation		_	_			
Video	https://www.yout	tube.com/wat	ch?v=55	5bI4AhA54		

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Charecteristics Adsorption

Isotherm

	The molecules of gases or liquids or the solutes in solutions adhere to the surface of the solids. In adsorption process, two substances are involved. One is the solid or the liquid on which adsorption occurs and it is called adsorbent. The second is the adsorbate, which is the gas or liquid or the solute from a solution which gets adsorbed on the surface.
	Adsorbent: The substance on whose surface the adsorption occurs is known as adsorbent.
Introduction	Adsorbate: The substance whose molecules get adsorbed on the surface of the adsorbent (i.e. solid or liquid) is known as adsorbate.
	Adsorption is different from absorption. In absorption, the molecules of a substance are uniformly distributed in the bulk of the other, whereas in adsorption molecules of one substance are present in higher concentration on the surface of the other substance.
	The type of interaction between the adsorbed molecule and the solid surface varies over a wide range from weak non-polar van der Waals' forces to strong chemical bonding. Examples of adsorption where ionic or covalent bonding occurs are the adsorption of chloride ions on silver chloride (ionic) or of oxygen gas on metals where oxygen-metal bonds are formed (covalent). In these cases the process is called chemisorption, and it is generally characterized by high heats of adsorption (from 10 to 100 Kcal per mole of gas adsorbed). Chemisorption is highly specific in nature and depends on the chemical properties of both the surface molecules and the adsorbed molecules. Adsorption arising from the weaker van der Waals' and dipole forces is not so specific in character and can take place in any system at low or moderate temperatures. This type of adsorption (less than about 10 Kcal/mole). The amount of solute adsorbed by a given quantity of adsorbent increases with the concentration of the solution. In some cases the layer of adsorbed molecules is only one molecule deep, and further adsorption stops when the surface of the crystal lattice is covered. The equilibrium between the dissolved solute and the material adsorbed also depends upon the nature of the solvent and the temperature, the amount adsorbed increasing at lower temperatures. From measurements at constant temperature, one can obtain a plot of x/m, the number of grams adsorbed per gram of adsorption isotherm.
Theory	(1) Freundlich isotherm : $x/m = kc^n$ (1) x = weight of material, in grams, adsorbed by m grams of adsorbing material c = concentration in solution ';;';klj =5TRF', mole liter n = constant ranging from 0.1 to 0.5 k = another constant Although k varies considerably with the temperature and nature of the adsorbent, the ratio of k values for two different adsorbents is constant for different solutions. By taking the logarithm of Eq. (1) we obtain log (x/m) = nlogc + logk (2) According to this equation a plot of 'log(x / m) versus logc' is a straight line, and the constants may be evaluated from the slope n and the intercept log k Langmuir isotherm :c
	$\frac{x}{m} = \frac{\alpha c}{1 + \beta c}$

	Or,
	$\frac{c}{(x/m)} = \frac{1}{\alpha} + \frac{\beta}{\alpha}c$
	α and β are constants. For cases in which this equation represents the data, $c/(x/m)$ may be plotted as a linear function of <i>c</i> , and the constants evaluated
	from the slope β/α and intercept $1/\alpha$ and are constants.
Objective	To investigate the adsorption isotherms from aqueous solutions of acetic acid on activated charcoal.

_ _ ____

Procedure	 Prepare six different concentrations of acetic acid solutions by pipetting separately 2, 5, 10, 20, 50, and 100 ml of 0.5N acetic acid into several volumetric flasks and diluting to 100 ml with distilled water. Each solution is transferred to a 250-ml Erlenmeyer flask, and 2 g of activated charcoal (weighed accurately to the nearest milligram) is then added to each flask. Blank determination (100 ml distilled water + 2 g of activated charcoal) should be made. Shake the Erlenmeyer flasks periodically for a period of 30 min and then allow it to stand at least 30 min for equilibrium. After equilibrium has been reached, each solution is filtered using suction filter. Pipette 25 ml solution from the clear supernatant solution and titrate with 0.25N NaOH using phenolphthalein as an indicator. Standardize 0.25N NaOH solution: Weigh accurately 0.5 g KHP into distilled water and titrate with NaOH, using phenolphthalein as an indicator. Standardize 0.5N acetic acid: Pipette 20 ml 0.5N acetic acid into an Erlenmeyer flask and titrate with 0.25N NaOH that has been standardized. 			
Observation Table	SI No.Final equm concn.The weight of acetic acid removed per gm. Of activated (x)The amount of acetic acid removed per gm. Of activated (x/m)1			
Calculation	 Plotx <i>m</i> against <i>c</i>. Test the applicability of the Freundlich isotherm by plotting log(x / m) against log<i>c</i>. Calculate constants <i>k</i> and <i>n</i> in Eq. (2). Test the applicability of the Langmuir isotherm by plotting <i>c</i> (x m) against <i>c</i>. Calculate constants α and β in Eq. (3). 			
Video	https://www.youtube.com/watch?v=E5qbNS0lH9k			

COURSE-Diploma in Chemical Engineering

SEMESTER-6th

CHEMICAL TECHNOLOGY LABORATORY



To determine the acid value of an given oil sample

Introduction	The acid value is defined as the number of milligrams of potassium hydroxide/
	sodium hydroxide required to neutralize the free fatty acids present in 1 gram of oil sample.
Theory	Fats are readily broken down by lipase into free fatty acids and glycerol during storage, particularly when the temperature and moisture contents are high. Fat hydrolysis gets accelerated due to microbial contamination. The acid value of a fat is the number of mg of KOH/ NaOH is required to neutralize the free acid in 1g of the oil. RCOOH+KOH → RCOOK+H ₂ O A known weight of oil sample is dissolved in a suitable solution and titrated with KOH/NaOH solution using phenolphthalein solution as indicator till pink color is obtained.
	Conical Flask
	Reagents: 1) 0.1 N NaOH
	2) Ethanol
	3) Phenolphthalein
	Apparatus: 1) Balance machine
	2) Burette with stand
	3) Measuring Cylinder
	4) Conical flask
	5) Hot plate
Objective	To determine the acid value of a given oil sample
Procedure	The whole procedure involves 4 steps.
	1) Chemical preparation
	2) Sample preparation
	4) Calculation
	1. Chemical preparation:
	a) Phenolphthalein indicator solution: Dissolve 2g Phenolphthalein indicator powder into 100 ml ethanol and mix well by shaking.
	b) 0.1N Sodium Hydroxide solution preparation : Dissolve 4g Sodium Hydroxide pellets into 900 ml of distilled water, cool and make a final volume of 1000 ml to standardize the solution before use.

	2. Sample preparation:				
	a) Take 10g of oil sample and note the sample weight.b) Measure 50 ml ethanol (99%) and pour it into a separate 150 ml conical flask				
	 c) Add 2-3 drops Phenolphthalein indicator solution and neutralize the ethanol by adding 0.1N NaOH drop-wise until light pink color solution is formed. 				
	d) Add the neutralize ethanol with the sample and shake to mix the solution.e) Heat and boil the solution until the sample is dissolved in the ethanol completely.				
	3. Titration:				
	a) Take 0.1N Sodium Hydroxide in the burette.b) Note the initial burette reading				
	 c) Start titration by adding few drops of phenolphthalein indicator solution. d) Titration should be continued with vigorous agitation. e) Pink color solution indicates the end point of the titration. f) Note the final burette reading. 				
Observ					
ation Table	Sample Weight (Ws)=				
	Normality of NaOH (N)= 0.1N				
	Volume of NaOH Solution (V) = Final Burette Reading- Initial Burette Reading				
	Molecular Weight of NaOH (MW_{NaOH})= 40.001				
Calculations	MW _{NaOH} * N *V				
	Acid Value = $\frac{Ws}{Ws}$				
	=				
Results					
	Acid Value=				
Remarks	The acid value of an oil sample has been checked successfully.				

Lab Manual	https://old.fssai.gov.in/Portals/0/Pdf/Draft_Manuals/OILS_AND_FAT.pdf
Experiment	https://www.youtube.com/watch?v=TaN1CimEqfY
al	
Graphics	
Relevant IS	
Code	
Quiz	Viva voce:
	1) What is Acid value?
	2) Define normality.
	3) What is the formula of acid value?
	4) Explain the procedure to find out the acid value of an oil sample.

COURSE-Diploma in Chemical Engineering SEMESTER-6th

CHEMICAL TECHNOLOGY LABORATORY



To determine the saponification value of an oil sample

Introduction	Fats and oils are composed of molecules known as triglycerides, which are esters composed of three fatty acid units linked to glycerol. Here, esters can be hydrolyzed in the presence of an acid, a base, or specific enzymes known as				
	lipase. So, the alkaline hydrolysis of ester is known as saponification which produces soap and glycerol.				
	Saponification value: The saponification value is the number of mg of potassium hydroxide required to hydrolyze or saponify 1 gram of oil/fat.				
Theory	In this process, triglycerides can be converted to soap by treated with a strong base which cleaves the ester bond, releasing fatty acid salts (soaps) and glycerol. Triglycerides are generally animal fats and vegetable oils. So, vegetable oils and animal fats are the traditional materials that are saponified. It is determined by measuring the alkali required to neutralize the free fatty acids by complete hydrolysis of l gm of oil/fat.				
	Oil /Fat + Base ———>Fatty acid salt (Soap) + Glycerol				
	The oil sample is saponified by refluxing with a known excess of alcoholic potassium hydroxide solution. The alkali required for saponification is determined by titration of the excess potassium hydroxide with standard hydrochloric acid. The saponification value gives an idea about the molecular weight of fat or oil. The long chain fatty acids found in fats have low saponification value because they have a relatively fewer number of carboxylic functional groups per unit mass of the fat and therefore high molecular weight. So, the smaller the saponification value means the higher the molecular weight. As the average molecular weight of oil or fat depends on the average length of carbon chain of the fatty acid components, the saponification value also gives an indication value for each oil has its own characteristic value.				
	Chemical Reaction:				
	$\begin{array}{cccccccccccccccccccccccccccccccccccc$				
	(Triglyceride) (Glycerol) (soap)				



- f) After 30 minutes, remove the reflux condenser and set a distillation unit.
- g) Distill and collect 180 ml ethanol after discarding first 10 ml.

- h) After that turn off the heating of the distillation unit.
- i) Now the collected neutralized ethanol is ready.
- j) Take KOH pellets in the mortar and grind them with the pastle.
- k) Take 6 g of ground KOH in a flask and add 150 ml collected neutralize ethanol.
- 1) Dissolve the KOH in ethanol by keeping the flask into cold water.
- m) Ethanolic Potassium Hydroxide Solution is ready now.

[B] 0.5 N Hydrochloric Acid:

Dilute 4.1 ml of Conc. HCL with distilled water to make a total volume of 100 ml.

[C] Phenolphthalein indicator solution:

Dissolve 2 g Phenolphthalein indicator powder into 100 ml of ethanol and mix well by shaking.

Step 2: Sample and Blank preparation

- a) Take two flasks of 250 ml for Sample and Blank preparation.
- b) Take 5 g oil/fat sample in the flask labeled with 'Sample'.
- c) Note the sample weight.
- d) Measure 50 ml of ethanolic KOH and pour into the sample flask.
- e) Measure 50 ml of ethanolic KOH again and pour into the Blank flask. No sample is there.
- f) Sample & Blank are ready. Separate layer of oil sample will be found in 'Sample' flask.

Step 3: Saponification

- a) For Saponification, place the sample flask on heating mantle.
- b) Attach the condenser with the sample flask.
- c) Heat the sample flask at the boiling point for 30 minutes.
- d) Turn on the cold water flow through the condenser.
- e) After 30 minutes of reflux, no separate layer will be found. The transparent and clear mixture indicates the end of the Saponification.
- f) In the same way boil and reflux the Blank for 30 minutes.

Step 4: Titration

[A] Titration of Sample:

- a) Add few drops of phenolphthalein indicator solution in the flask and shake the flask for proper mixing.
- **b)** Take 0.5 N HCL solutions in a burette.
- c) Note the initial burette reading.
- d) Start titration with vigorous agitation of the flask.
- e) Stop titration when the pink color is disappeared completely.
- f) Note the final burette reading.

	[B] Titration of Blank Sample:
	 a) After refluxing, cool the sample completely and bring for titration. b) Add few drops of phenolphthalein indication solution into the blank sample and shake the flask for proper mixing. c) Take 0.5 N HCL solutions in a burette. d) Start titration after taking the initial burette reading. e) Titration should be carried out with vigorous agitation of the flask. f) Stop the titration when pink color is disappeared completely. g) Note the final burette reading for blank sample.
Observ ation Table	Sample Weight (Ws) = Burette reading :
	For Sample (V _S): Final reading – Initial reading =
	For Blank Sample (V _B): Final reading – Initial reading =
Calculations	Saponification Value = $\frac{56.1 \times (V_B - V_S) \times N}{W_S}$
	= molecular weight of KOH =56.1 N= Normality of standard HCL Solution = 0.5
Results	Saponification Value=
Remarks	 The Saponification value of an oil sample has been checked successfully. PRECAUTIONS: Do not touch KOH without gloves as it may burn the skin. Do not breathe the fumes of KOH. Please ensure that all glassware like is Burette, Conical flask etc. are clean and free of any impurities. Carry out the titration most accurately
Lab Manual	https://old.fssai.gov.in/Portals/0/Pdf/Draft_Manuals/OILS_AND_FAT.pdf
Experiment	https://www.youtube.com/watch?v=DpgmHx-dl1A
al Graphics	
Relevant IS	
Code	Vivo vogo:
	1. What is Saponification value?
	2. What is the formula of Saponification value?
	3. Explain the procedure to find out the saponification value of an oil sample.

COURSE-Diploma in Chemical Engineering SEMESTER-6th

CHEMICAL TECHNOLOGY LABORATORY



Standardization of Sodium Thiosulphate using Potassium Dichromate Solution

Introduction	'Sodium Thiosulphate' is a very important industrial raw material. It is widely used in photographic studio. The objective of this experiment is to determine the strength of this Sodium Thiosulphate. This experiment is based on oxidation and reduction reaction, as well as "Iodometric reaction". Titration involving with iodine or dealing with iodine liberated in chemical reaction is called Iodimetric and Iodometric titration respectively. This reaction is iodometric because iodine is got from KI. The main purpose of the experiment is to standardize Na ₂ S ₂ O ₃ solution with standard K ₂ Cr ₂ O ₇ solution.
Theory	Sodium thiosulphate solution is standardized against potassium dichromate in presence of hydrochloric acid and potassium iodide. Potassium dichromate oxidizes the iodide ion in acidic medium to equivalent amount of iodine. The iodine formed in the reaction oxidizes sodium thiosulphate giving sodium tetrathionate ion and the end point is detected by starch solution. $K_2Cr_2O_7 + 6KI + 14HCI \longrightarrow 3I_2 + 2CrCI_3$ (Pot. Dichromate)(Pot. Iodide)(Hydrochloric acid) (Iodine) (Chromium chlorate) + 8KCI + 7H_2O (Pot. Chloride) (Water) 2Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI (Sod. thiosulphate) (Iodine) (Sod. tetrathionate) (Sod. iodide) Here potassium dichromate (K_2Cr_2O_7) is an oxidizing agent and Iodide (I ⁻) is a reducing agent. Again in the second reaction Iodine (I_2) is an oxidizing agent and thiosulphate (S_2O_3 ²⁻) is a reducing agent. In the 2nd Step of the reaction a specific indicator is used that is "Starch"- which has a significant characteristic on iodine. Starch + I_2 = Starch-Iodine (blue color) Starch-Iodine + 6S_2O_3 ²⁻ = Starch + 6I ⁻ + 3S_4O_6 ²⁻ If the solution contains a little iodine, some starch solution is added and Na_2S_2O_3 is run in from the human the human of the starch iodine anomalor will
	Is full in from the outere, the oute color of the statch-found complex will disappear from the solution as soon as all the iodine has been reduced to iodide ion.Hence, based on the above theory our aim is to prepare and standardize sodium thiosulphate using potassium dichromate and potassium iodide in presence of hydrochloric acid and starch solution as indicator. This experiment is very important because it gives the idea of oxidation and reduction reaction and at the same time, it views a clear idea about iodometric reaction.
Objective	Standardization of sodium thiosulphate using potassium dichromate solution.

Procedure	The whole procedure involves 5 steps:					
	The whole procedule involves 5 steps.					
	 Chemical and reagent preparation Sodium Thiosulphate solution preparation 					
	3. Potassium Dichromate solution preparation					
	 Sodium Thiosulphate solution standardization Calculation 					
	Apparatus required:					
	1. Balance Machine 2. Duratte with Stand					
	3. Hot plate					
	4. Conical flask					
	5. Volumetric flask 6. Measuring cylinder					
	7. pipette					
	Chemicals required:					
	 Concentrated Hydrochloric Acid (37%) Potassium Dichromate (99 5%) 					
	 Sodium Thiosulphate (Pentahydrate) (99%) 					
	4. Potassium Iodide					
	5. Starch Powder					
	Step 1: Chemical and reagent preparation					
	[A] 1% Starch Solution:					
	a) Measure 50 ml distilled water and take into a beaker.					
	b) Place the beaker on a hot plate and apply heat to boil the water c) Weight 0.5g starch powder and transfer it into the hot water					
	d) Stir the content using a glass rod periodically during heating.					
	e) Fold a filter paper and filter the solution when the starch is dissolved					
	f) Transfer the filtered 1% starch solution into a suitable container.					
	g) 1% starch solution is ready to use in the test as indicator.					
	[B] 15% Potassium Iodide Solution Preparation:					
	a) Dissolve 15g Potassium Iodide (KI) crystal into 100 ml distilled water.					
	 b) Stir to dissolve the Potassium Iodide crystal completely. c) Store in dark and use immediately after preparation 					
	c) store in dark and use inimediately after preparation.					
	Step 2: 0.1M Sodium Thiosulphate Solution Preparation:					
	a) Weight 2.48g Sodium Thiosulphate crystal.					
	b) Transfer the Sodium Thiosulphate crystal in a volumetric flask of 100 ml.					
	d) Attach the cap to the flask and swirl it to dissolve the crystal in water.					
	e) Label the flask as "Expected 0.1 M Sodium Thiosulphate".					

	Step 3: Potassium Dichromate Solution Preparation:
	 a) Take a portion of Potassium Dichromate crystals in a glass dish. b) Spread the Potassium Dichromate crystals on the surface of the glass dish. c) Dry the Potassium Dichromate crystals in a hot air oven at 110°C for 30 minutes. d) After 30 minutes of drying, take out the dish from oven and cool in desiccators for 10 minutes. e) After 10 minutes of cooling, take weight of 0.16-0.22g dried Potassium Dichromate crystals. f) Note the weight of Potassium Dichromate. g) Transfer the Potassium Dichromate into a conical flask. h) Measure 25ml distilled water and pour into the flask. i) Swirl the flask to dissolve the Potassium Dichromate in water completely. j) Now, add 5ml concentrated hydrochloric acid in to the flask drop wise. k) Measure 20 ml of 15% Potassium Iodide solution and pour into the flask. l) Swirl the flask to mix the content properly and wait for 5 minutes.
	n) Swirl the flask to mix the content properly.
	Step 4: Sodium Thiosulphate Solution Standardization:
	 a) For standardization, take the newly prepared sodium thiosulphate solution in a burette. b) Note the initial burette reading. c) Titrate the potassium dichromate solution by sodium thiosulphate. d) Carry out the titration until radish brown color is almost disappeared. e) Pause the titration when yellow color is fade out. f) Now, add 1 ml of 1% starch solution into the flask. g) Swirl the flask to mix the content properly. h) Resume the titration in the same way as done before. i) Titration should be carried out with vigorous agitation of the flask. j) Stop the titration when the dark blue color is fade out to sea green color. k) Note the final burette reading.
Observ ation Table	Weight of Potassium Dichromate (W) =
	= Final Burette Reading – Initial Reading =
Calculations	$M = \frac{20.394^* \times W}{V}$
	=
	*20.394 is a calculation factor given by AOAC official method.

Results	Molarities of Sodium Thiosulphate (M) =
Remarks	The Molarity of Sodium Thiosulphate solution has been checked successfully.
Lab Manual	https://kb.psu.ac.th/psukb/bitstream/2553/1406/3/243342_app.pdf
Experiment al Graphics	https://www.youtube.com/watch?v=X2KrBfnKw24
Relevant IS	
Quiz	 Viva voce: 1. How do you standardize sodium thiosulphate using potassium dichromate? 2. Why this titration is called Iodometric titration? 3. Why iodine solution is is kept in dark? 4. Why is the starch indicator added close to the end point?

COURSE-Diploma in Chemical Engineering SEMESTER-6th

CHEMICAL TECHNOLOGY LABORATORY



Determination of iron in a sample of cement

Introduction	Cement, as a binding material, is a very important building material. Almost every construction work requires cement. Therefore, the composition of cement is a matter of great interest to engineers. A simple, rapid and sensitive spectrophotometric method is developed for the determination of trace amounts of iron. The proposed method is based on the reaction of iron with ammonium thiocyanate forming a complex having a maximum absorption at 450 nm. Iron is estimated colorimetrically and the method is economical and less time consuming. The method is successfully applied to cement sample and results is found to be accurate and precise as the more sophisticated colorimeter is commonly used for ferrous determination.			
Theory	For understanding cement composition Cement ingredients. By altering the production, one can achieve the desired There are seven major ingredients of ingredients in cement is given below.	on, one must know the functionality of amount of an ingredient during cemen cement quality. cement. The general percentage of thes	of nt se	
	Ingredients	Percentage in cement		
	Lime	60-65		
	Silica	17-25		
	Alumina	3-8		
	Magnesia	1-3		
	Iron oxide	0.5-6		
	Calcium Sulfate	0.1-0.5		
	Sulfur Trioxide	1-3		
	 Iron oxide: Chemical formula is Fe₂O₃ Iron oxide imparts color to cement. It acts as a flux. At a very high temperature, it imparally aluminum to form tricalcium alumi Tricalcium alumino-ferrite imparts 	rts into the chemical reaction with calcium an no-ferrite. hardness and strength to cement	ıd	
	Colorometric measurements were made usi 450 nm .The basis of spectrophotometric m absorption of radiation by the solution and When monochromatic light passes through rate of decrease in intensity with the co- directly proportional to the intensity of t analyte in the solution spectrophotometric complex. Ammonium thiocyanate yields a colour produced is stable in nitric acid med certain colouring reagent and optical dens 450 nm since the maximum absorption was at 450 nm. Its optical density is measured in found from the standard calibration curve.	ng a colorimeter and samples were analysed nethods is the simple relationship between the 1 the concentration of species in the solution a transparent medium (coloured solution) the ncentration and thickness of the medium he light. In order to determine a species of cally, it is usually converted into a colore a blood red colour with ferrous iron and the ium. The sample of cement was analysed with ity of the coloured compound is measured at 450nm, hence all measurements were made n a colorimeter and the concentration of iron	at ne n. is or ed ne th at de is	

Objective	To estimate the amount of iron present in the given sample of cement by
	colorimetric using ammonium thiocynate as the reagent.
Procedure	Apparatus required:
	 Beakers std flask, Watch glass, Colorimeter
	Chemicals required: 1. Cement 2. Concentrated Hydrochloric Acid (HCL) 3. Nitric acid (HNO ₃)
	Methodology:
	Step 1: Preparation of Standard Curve
	 Dissolve the given ferrous ammonium sulphate (0.7022 g) in 100 ml of water and add 5 ml of 1 : 5 H₂SO₄ followed by dil. KMnO₄ solution through burette until light-pink color appears. Dilute the solution to 1 L such that 1 ml of solution contains. 0.1 mg of Fe⁺². From the above solution, take separately 1, 2, 3, 4, 5 ml into separate 100 ml standard volumetric flasks. Add 1 ml of nitric acid and 5 ml of 40% ammonium thiocyanate solution to all the above samples to get blood red color. Make up the solutions to the mark by adding distilled water. Now measure the optical densities of all the solutions using colorimeter table 1 and plot a graph by taking amount of ferrous iron on x-axis and optical density on the y-axis. The curve obtained is called standard calibration curve in figure 1.
	Steps 2: Dissolution of sample
	 Weigh out accurately about 0.1 g of the given cement sample into a clean 250 ml beaker. Add about 5 ml water and stir with a stirrer. Add few drops of concentrated HCl through the walls of the beaker and stir again. Heat the mixture until the moisture is evaporated. Then add 20 ml of distilled water and dissolve the content and make up the solution with distilled water to 100 ml. Shake well for uniform concentration.
	Steps 3: Development of color
	 Pipette out 10 ml of the solution prepared above in to 100 ml std. flask. Add 1 ml of concentrated HNO₃.

3. Add 5 ml of 40% ammonium thiocynate (NH₄SCN) with a Burette.

	4. 5. 6.	Make u concent Now tal (optical From th curve.	up the solution up ration. the sample solution density) using the pl and O.D. find out th	to the m on into colo noto colorir e concentra	ark and sorimeter tu neter. ation of F	shake well for be and measure t e ²⁺ from the cal	uniform he O.D. libration
Calculations	W1=W W2=W W1-W	W1=Weight of the weighing bottle cement W2=Weight of the weighing bottle W1-W2=Weight of the cement sample					
Observ ation	Table	1 Ontica	l density values for s	tandard Ca	libration (urve ·	
Table	Tuore	<u>si Na</u>	Concentration of	E +2 :			-
		51. INO.	Concentration of	re in mg		OD	
		1.	0.2			0.18	
		2.	0.4			0.39	
		3.	0.6			0.58	-
		4.	0.8			0.81	-
		5.	1			0.98	-
	Absorbance	1 0.8 0.6 0.4 0.2 0 0	0.2 0.4 Am	0.6 ount of Fe	0.8 errous iron	1 1 1	
			Fig. 1. Sta	ndard Calibra	tion curve	g	

Results	
	Concentration of Fe ⁺² in mg = $\underline{y * 10* 100}$ W1-W2
	y = amount of ferrous iron from the calibration curve
Remarks	The concentration of ferrous iron has been checked successfully.
Lab Manual	https://www.jetir.org/view?paper=JETIR1812B71
Experiment al Graphics	
Relevant IS Code	
Quiz	 Viva voce: 1. What are the chemicals are used in this experiment? 2. What are the apparatus are used in this experiment? 3. Cement composition. 4. Molecular weight of HCl.

COURSE-<mark>Diploma in Chemical Engineering</mark> SEMESTER-<mark>6th</mark>

CHEMICAL TECHNOLOGY LABORATORY



Determination of percentage of available chlorine present in Bleaching powder
Introduction	Bleaching powder, also known as chlorinated lime, is a yellowish-white powder having a smell of chlorine and is readily soluble in water. Besides bleaching action chlorine has got strong germicidal and disinfectant properties also. Accordingly, it finds application as a disinfectant for drinking water or swimming pool water. Industrially, the bleaching powder finds major use in chemical, paper, textile and oil industries. The bleaching, oxidizing or disinfecting potential of a sample of bleaching powder depends on the percentage of chlorine liberated on action of acid. We may define available chlorine to be the grams of chlorine liberated from 100 g of the bleaching powder on treatment with dilute acid. In this experiment iodide ions (from KI) are used to determine available chlorine in bleaching powder. Such a determination wherein iodide ions are used as a reducing agent is termed as iodometric determination.
Theory	Bleaching powder is prepared by passing chlorine gas over slaked lime at a
	Bleaching powder is prepared by passing chlorine gas over slaked lime at a temperature of 35-45°C and consists of a mixture of calcium hypochlorite Ca (OCl ₂) and calcium chloride CaCl ₂ ; in addition some amount of free slaked lime i.e. Ca(OH) ₂ .H ₂ O is generally present. Of these, Ca (OCl ₂) is responsible for the bleaching action of bleaching powder. Due to its hygroscopic nature, bleaching powder absorbs moisture from atmosphere and evolves chlorine. Due to this deterioration, a sample of bleaching powder may always contain lesser amount of chlorine than expected and therefore a sample of bleaching powder needs to be analyzed for its effective or available chlorine. A sample of bleaching powder liberates chlorine gas (Cl ₂) on treatment with glacial acetic acid, as per the following reaction.
	$CaOCl_2 + 2 CH_3COOH \rightarrow (CH_3COO)_2 Ca + H_2O + Cl_2 \uparrow \dots \dots (1)$
	The amount of chlorine liberated by the action of an acid on bleaching powder (CaOCl ₂) is termed as available chlorine. The liberated chlorine can be used to oxidize KI (taken in excess) in presence of acid and liberate out an equivalent amount of iodine as per the following equation: $Cl_2 + 2KI \longrightarrow 2KCl + I_2(2)$
	This iodine can then be determined by titrating against a standardized solution of sodium thiosulphate using freshly prepared starch solution as an indicator. The chemical reactions involved can be given as follows: $I_2 + 2 Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6(3)$
	The overall reaction between the chlorine liberated from the bleaching powder and sodium thiosulphate mediated by potassium iodide can be obtained by adding eqn. (2) and (3).
	$Cl_2 + 2KI \longrightarrow 2KCl + I_2$
	$12 + 2 \operatorname{INa2S2O3} \rightarrow 2\operatorname{INa1} + \operatorname{INa2S4O6}$ $2 \operatorname{Na2S2O3} + \operatorname{Cl}_2 + 2\operatorname{KI} \rightarrow \operatorname{Na2S4O6} + 2\operatorname{NaI} + 2\operatorname{KCI}$
	The sodium thiosulphate can be standardized by titrating against a primary standard solution of potassium dichromate.

Objective	Determination of percentage of available chlorine present in Bleaching powder
Procedure	Apparatus required: 1. Burette 2. Pipette 3. Measuring flask 4. Conical flask 5. Funnel 6. Mortar and pastle Chemicals required: 1. bleaching powder 2. Potassium Iodide 3. Glacial Acetic Acid 4. N/10 sodium thiosulphate solution 5. Starch Solution Methodology: Step 1: Preparation of Bleaching Powder Solution 1. Weigh out accurately 3 g of bleaching powder in a cleaned and pre-weighed weighing bottle. 2. Transfer it into a mortar, crush with a pestle and little distilled water. 3. Make a thin paste of bleaching powder sample with distilled water. 4. Transfer the paste into 250 ml volumetric flask. 5. Wash the mortar and pestle with distilled water and transfer it into the flask to make 250ml. 6. Shake well to get homogenous suspension of bleaching powder. Steps 2: Estimation of Available Chlorine 1. Wash the burette with distilled water and rinse with standard hypo solution then fill the burette. 2. Pipette out 10 ml of homogenous solution of bleaching powder in the 20 ml conical flask. 3. Put 4ml of potassium iodide (KI) and about half test tube of glacial acetic acid into the flask. 4. Titrate the liberated iodine against (N/10) standard hypo sol

Observ					
ation Table	Valaria a f		Burette Reading (ml)		Volume of Hypo
	SI. No	Volume of Bleaching Powder solution V1 (ml)	Initial Reading	Final Reading	Solution Consumed (ml)
	1	10			
	2	10			
	3	10			
Calcul ation	Bleaching Powder Vs Sodium thiosulphate solution				
	$\mathbf{N}_1 \mathbf{V}_1 \qquad = \mathbf{N}_2 \mathbf{V}_2$				
		Ν	1 × 10 =	$= 1/10 \times V_2$	
		Ν	N1 =	$= V_2/100$	
	Strength of available chlorine $= N_1 \times 35.5$ (Eq. Wt of chlorine) g/l				
		% of available chlorin	e =	$= \frac{N_1 \times 35.5 \times 25}{1000 \times V}$	0×100 V
		W= amount of bleachi	ng powder		
Results	Amount	of available chlorine pr	esent in bleac	ching powder is	5 %
Remarks	The per checked	centage of available chl successfully.	orine present	in Bleaching p	owder has been
	PRECAUTIONS:				
	 Wash Samp the atmo Rinse The r Titrat libera 	the glass apparatus with the of bleaching powder osphere. the burette with standar eagent must be prepared tion must be started imm ted instantly.	h the distilled must not be k rd N/10 sodiu l freshly. nediately after	water. ept open as it a m thiosulphate the addition of	bsorbs moisture from solution. f acid because chlorine
Lab Manual	https://w	ww.dbit.ac.in/applied-scie	ences/syllabus/	chemistry-lab.pc	l <u>f</u>

Experiment al Graphics	https://www.youtube.com/watch?v=vqXBu0sSbQA
Relevant IS	
Code	
Quiz	 Viva voce: 1. Define 'available chlorine' in bleaching powder. 2. Why the 'available chlorine' in bleaching powder decreases with time? 3. What happens when chlorine is bubbled through potassium iodide solution?

COURSE-Diploma in Chemical Engineering SEMESTER-6th

CHEMICAL TECHNOLOGY LABORATORY



Determination of percentage of available oxygen in pyrolusite

Introduction	
Introduction	
	Manganese does not occur free in nature. Its most commonly found ore is
	Pyrolusite (MnO ₂) which sometimes contains small amounts of Brounite
	(Mn ₂ O ₃), Manganite [Mn ₂ O ₃ ,H ₂ O or MnO(OH)], Hausmannitie (Mn ₃ O ₄) and
	some other impurities. When the ore is to be used for metallurgical purpose (i.e.
	as a source of manganese metal or for the production of ferromanganese), it is
	graded on the basis of its manganese content which can be determined by
	dissolving the ore in nitric acid and oxidizing manganese to permanganic acid
	using sodium bismuthate (NaBiO ₃). After filtering off the excess of the
	bismuthate, the permanganic acid is determined with a standard ferrous ion
	solution.
	Being an efficient oxidizing agent pyrolusite is directly put to several industrial
	use such as:
	i) Production of Cla by the action of some HCl (Woldon process)
	i) A drive in points
	11) A drier in paints.
	111) A depolarizer in Leclanche cell and Dry cell
	iv) Bleaching Agent
	v) A catalyst in the preparation of oxygen by heating potassium chlorate
	(KClO ₃).
	For these purposes, the ore is graded on the basis of its oxidizing capacity which
	is reported in terms of the percentage of 'Available Oxygen' in pyrolusite.
	'Available oxygen' of pyrolusite is that part of oxygen which is available for
	oxidation of a reducing agent when the ore is treated with strong acid.
	$MnO_2 + 2H^+ \longrightarrow Mn^{2+} + H_2O + O$
	(available oxygen)
Theory	
	A known weight of dry ore is heated with conc. HCl. Chlorine equivalent to
	available oxygen is liberated as per the following equation:
	$MnO_2+4H^++2e \longrightarrow Mn^{2+}+2H_2O$
	$2Cl^{-} \longrightarrow Cl_{2} + 2e$
	$MnO_2 + 4H^+ + 2Cl^- \longrightarrow Mn^{2+} + Cl_2 + 2H_2O$
	Or $MnO_2 + 4HC1 \longrightarrow MnCl_2 + Cl_2 + 2H_2O$
	The chlorine gas thus evolved is passed into a solution of KI when an equivalent
	amount of iodine is liberated
	$C_{1}^{1} + 2_{2}^{2} \rightarrow 2C_{1}^{1-1}$
	$C_{12} \pm 2c$ \rightarrow $2C_1$
	$21 \longrightarrow 1_2 + 2e$
	$Cl_2 + 2l \longrightarrow l_2 + 2Cl^-$
	which is titrated with a standard solution of sodium thiosulphate using starch
	solution as indicator near the end-point.

	$I_2 + 2e \longrightarrow 2I^-$ $2S_2O_3^{2-} \longrightarrow S_4O_6^{2-} + 2I^-$
	The amount of thiosulphate consumed corresponds to the oxygen available from pyrolusite.
Objective	Determination of percentage of available oxygen in pyrolusite
Procedure	 Reagents required: 1. Standard sodium thiosulphate solution (N/10) 2. Potassium iodide solution (5%) 3. Freshly prepared starch solution 4. Conc. HCl
	Place about 100 ml of a 5% solution of KI (AR Grade) in the conical flask. Weigh accurately the sample bottle containing the finely pulverized and dry pyrolusite ore. Transfer about 0.5g of the sample to the distillation flask and weigh the bottle again. Add about 30 ml of conc. HCl to the flask. Heat the distillation flask gently and from a Kipp's apparatus, pass a slow and steady stream of CO ₂ into the contents of the distillation flask. After the ore has completely dissolved, raise the temperature and boil the contents for about 10 minutes. Now, disconnect the delivery tube from the distillation flask. Rinse the conical flask and make up the volume up to the mark. Transfer 50 ml of this solution on to a conical flask and titrate against 0.1N Na ₂ S ₂ O ₃ , when the colour of the solution turns straw yellow, add about 2 ml of freshly prepared starch solution and titrate further till the blue colour just disappears. Take the initial and final titration readings.
Observ ation Table	Initial weight of sample bottle = w1 g Final weight of sample bottle = w2 g Therefore, weight of pyrolusite sample taken = (w1-w2) g Total volume of iodine solution prepared = 250 ml Volume taken for each titration = 50 ml

a b b b	
Calculations	Let the concordant volume of 0.1 N $Na_2S_2O_3$ used = A ml
	Then, $N1 V1 = N2V2$ (Iodine Solution) $(0.1 N Na_2S_2O_3)$
	$N1 \times 50 = \frac{1}{10} \times A$
	N1 = A 10×50
	i)Strength of iodine solution in terms of $MnO_2 = A \times 43.47 \text{ g/l}$ 10×50
	Or, weight of MnO ₂ in (w1-w2)g of pyrolusite = $\underline{A} \times \frac{43.47 \times 250}{1000}$ g
	Therefore, % of MnO ₂ in pyrolusite = $\frac{A \times 43.47 \times 250}{10 \times 50 \times 1000} \times \frac{100}{\text{w1-w2}}$
	$= \underline{A \times 43.47}_{20 \times (w1-w2)}$
	ii) Strength of iodine solution in terms of available oxygen = $\underline{A \times 8}_{10 \times 50}$ g/l
	Weight of oxygen available from (w1-w2) g pyrolusite = $A \times 8 = \frac{A \times 8}{10 \times 50} \times \frac{250 \text{ g}}{1000}$
	% Available oxygen = $\underline{A \times 8}_{10 \times 50} \times \underline{250}_{1000} \times \underline{100}_{w1-w2}$ g
	$= \underline{A \times 8}_{20 \times (w1-w2)}$
Results	% Available oxygen =
Remarks	The percentage of available oxygen in pyrolusite has been checked successfully.
	 Precautions: To ensure complete and rapid decomposition of the ore by conc. HCl. To avoid the absorption of moisture by the powdered ore, it should be immediately transferred to a small stopper weighing bottle. To make the apparatus airtight, all connections should be made with ground glass joints. CO2 should be passed at a very slow rate otherwise some iodine might be lost from the solution. Before discontinuing heating at the end, disconnect the delivery tube to avoid back-suction of the absorption solution.

Lab Manual	Applied Chemistry : Theory And Practice. By O. P. Vermani
Experiment	
al	
Graphics	
Relevant IS	
Code	
Quiz	Viva voce:
	1. Define 'Available oxygen' in pyrolusite?
	2. Why we determine 'available oxygen' in pyrolusite?
	3. What are the uses of pyrolusite in industries?
	4. Explain the procedure to find out the 'available oxygen' in pyrolusite.

COURSE-Diploma in Chemical Engineering SEMESTER-6th

CHEMICAL TECHNOLOGY LABORATORY



To determine the iodine value of an oil

Introduction	
	Iodine value or iodine number is the number of grams of iodine consumed by
	100g of fat.
	The lodine value is used to measure the amount of un-saturation of oils and fats.
	A higher todine value indicated a higher degree of un-saturation. There are
	them is determining the indine value of fate
Theory	them is determining the fourie value of fats.
Theory	Animal or vegetable oils and fats known chemically as triglycerides that composed of three fatty acid units linked to glycerol. Fatty acids are long chain hydrocarbon with carboxyl (COOH) group which can be saturated or unsaturated depending on the double bond between carbon atoms. Saturated fatty acids contain only single bond between carbon atoms and are tend to be solids at room temperature. Unsaturated fatty acids contain double bond between carbons atoms in addition to the single bonds present in the fatty acid chain. They are likely to exist as liquid at room temperature. Triglycerides with one double bond are known as mono-unsaturated and those with more than one double bond are known as poly-unsaturated. Depending upon the degree of un-saturation the fatty acids can be combined with oxygen or balogens to form saturated fatty acids
	can be combined with oxygen or halogens to form saturated fatty acids.
	The oil/fat sample is taken in carbon-tetrachloride and is treated with a known excess of iodine monochloride solution or Wij's solution. Iodine can combine with fats that have carbon double bonds and therefore the number of such bonds can be deduced from the amount of iodine with which they will combine. H H
	$>C=C < + ICl \longrightarrow -C -C - C - I - I - I - I - I - I - I -$
	After the reaction is complete, the amount of iodine that has reacted is determined
	by adding a solution of polassium lodide to the reaction product.
	$ICl + KI \longrightarrow KCl + I_2$
	This cause the remaining unreacted ICl to form molecular iodine. The liberated I_2 is than titrated with a standard solution of 0.1 N Sodium Thiosulfate.
	$I_2 + 2 Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$
	Unsaturated fat, which include mono-saturated and poly-saturated fats, have the reputation of being healthier than saturated fats, the main health benefit of unsaturated fatty acid is its ability to help lower the cholesterol and triglyceride levels (triglyceride is most common fat in our body and indicates high cholesterol levels and heart problems). The degree of iodine value indicates that the content of unsaturated triglyceride in certain fatty acid. Thus it indicates that which oil is

healthier.

Objective	To determine the iodine value of an oil.
Procedure	The whole procedure involves 4 steps:
	 Chemical preparation Sample and Blank preparation Titration Calculation
	Apparatus required:
	 Balance Machine Burette with Stand Measuring Cylinder Conical Flask Pipette Funnel Beaker Hot Plate
	Chemicals required:
	 Starch Soluble Potassium Iodide Sodium Thiosulfate Carbon Tetrachloride Wij's Solution
	Step 1: Chemical preparation
	[A] 1% Starch Solution:
	 a) Measure 50 ml distilled water and take it into a 100 ml glass beaker. b) Place the beaker on a hot plate and boil the water. c) Take weight of 0.5g starch soluble and transfer it into the boiling water. d) Stir the starch solution to dissolve it in water. e) Clear and transparent solution indicates that the starch is dissolved completely. f) Prepare a filter paper and place it on a funnel. g) Now filter the hot starch solution. h) After 30 minutes, collect the filtrate. i) 1% starch solution is ready for using it in the titration as an indicator.
	[B] 0.1N Sodium Thiosulfate solution:
	 a) Take weight of 2.5g Sodium Thiosulfate crystal. b) Label a volumetric flask of 100 ml with "0.1 N Sodium Thiosulfate". c) Fill the flask with 80 ml distilled water. d) Transfer the measured Sodium Thiosulfate crystal into the flask. e) Shake the flask to dissolve the chemical in water. f) Heat the flask for 10 minutes to dissolve Sodium Thiosulfate completely.

g) After 10 minutes, turn off heating and cool the flask at room temperature.

- h) After cooling add water to make the final volume of 100 ml.
- i) Standardize the solution before use.

Step 2: Sample and Blank preparation

- a) Take 2.7 g of oil sample into a conical flask.
- b) Note the sample weight.
- c) Prepare a Blank to another conical flask without taking sample into it.
- d) Pipette 25 ml carbon tetrachloride and pour into the sample flask.
- e) Close the flask with its stopper immediately.
- f) Pipette another 25 ml carbon tetrachloride again and pour into the Blank flask. Close the flask with its stopper immediately.
- g) Pipette 25 ml Wij's solution and pour into the sample flask and close the flask with its stopper immediately.
- h) Similarly, pipette 25 ml Wij's solution and pour into the Blank flask and close the flask with stopper immediately.
- i) Shake and rotate both flasks together for proper mixing.
- j) Add potassium iodide crystal on all round surface of the stopper.
- k) Keep both flasks in dark for 30 minutes.

Step 3: Titration

[A] Titration of Sample:

- a) Take standardized 0.1N Sodium Thiosulfate solution in a burette.
- b) Note the initial burette reading.
- c) Measure 100 ml distilled water.
- d) Pour the water into the sample flask washing the stopper into the flask.
- e) Shake and rotate the flask for proper mixing.
- f) Measure 1 ml 1% starch solution and keep ready to use later.
- g) Start titrating using 0.1N Sodium Thiosulfate.
- h) Add 1% starch solution when the solution color is changing to lighter.
- i) Shake the flask and resume the titration.
- j) Milky white color solution indicates the end point of the titration.
- k) Shake the flask vigorously. If the blue color comes back, titrate again.
- 1) Titration of sample is done.
- m) Note the final burette reading.

[B] Titration of Blank Sample:

- a) Start the titration of Blank in the same manner as like as sample titration.
- b) Note the initial burette reading.
- c) Add 100 ml distilled water washing the stopper and potassium iodide into the flask.
- d) Shake and rotate the flask for proper mixing.
- e) Measure 1 ml 1% starch solution and keep ready to use later.
- f) Start titration using 0.1N Sodium Thiosulfate.
- g) Add 1% starch solution when the solution color is changing to lighter.
- h) Shake the flask and resume the titration.
- i) Milky white color solution indicates the end point of the titration.
- j) Shake the flask vigorously. If the blue color comes back, titrate again.
- k) Titration of Blank is also done.
- l) Note the final burette reading.

Observ ation Table	Sample Weight (Ws) = Normality of Sodium Thiosulfate (N)= 0.1N
Calculations	
Surveilations	$12.69 \times (V_B - V_S) \times N$
	Iodine Value =
	=
	Calculation factor = 12.69 (given by AOAC official method) N= Normality of standard Sodium Thiosulfate solution
Results	Iodine Value=
Remarks	 The Iodine value of an oil sample has been checked successfully. Precautions: Iodine monochloride is caustic. So handle the reagent with gloves. For better results, performs the experiment without any time gap during addition of reagents as the liberated iodine is susceptible to oxidation by light.
Lab Manual	https://old.fssai.gov.in/Portals/0/Pdf/Draft_Manuals/OILS_AND_FAT.pdf
Experiment al	https://www.youtube.com/watch?v=fQ1hSNGnXYY
Graphics	
Relevant IS	
Quiz	Viva voce:
-	1. What is Iodine value?
	2. Why we use KI in determination of iodine value?
	3. Explain the procedure to find out the iodine value of an oil sample.

SEMESTER- 6th

<u>PETROCHEMICALS ENGINEERING</u> <u>LABORATORY</u>

EXPERIMENT-1

Preparation of Acid catalysed Phenol formaldehyde resin

Phenol-formaldehyde resin , also called phenolic resin , any of a number
of synthetic resins made by reacting phenol (an aromatic alcohol derived from benzene) with formaldehyde (a reactive gas derived from methane). Phenol formaldehyde resins (PFs) are condensation polymers and are obtained by condensing phenol with formaldehyde in the presence of an acidic or alkaline catalyst. They were first prepared by <i>Backeland</i> , an American Chemist who gave them the name as <i>Bakelite</i> . These are thermosetting polymers.
Acid catalyzed phenol-formaldehyde resin synthesis process is a two-step process. In the 1 st step, Novolac is produced as an intermediate followed by crosslinked polymer or Bakelite is produced. Novolac (or novolak), a solid prepolymer which resembles the final polymer except that it is of much lower molecular weight and is still thermoplastic (that is, it can be softened by reheating without undergoing chemical decomposition). Curing can be accomplished by grinding the novolac to a powder, mixing it with fillers such as wood flour, minerals, or glass fibres, and then heating the mixture in a pressurized mold. In order to be cured to a thermosetting resin, novolacs require the addition of more formaldehyde or, more commonly, of compounds that decompose into formaldehyde upon heating.
Thermosets: - The polymers which on heating change irreversibly into hard rigid and infusible materials are called thermosetting polymers. These polymers are usually prepared by heating relatively low molecular mass, semi fluid polymers, which becomes infusible and form an insoluble hard mass on heating. The hardening on heating is due to the formation of extensive cross-linking between different polymeric chains. This leads to the formation of a three dimensional network of bonds connecting the polymer chains. Since 3D network structure is rigid and does not soften on heating, the thermosetting polymers are Urea-Formaldehyde resin and Phenol- Formaldehyde (Bakelite) resins.
 Properties:- Phenol- formaldehyde resins having low degree of polymerization are soft. They possess excellent adhesive properties and are usually used as bonding glue for laminated wooden planks and in varnishes and lacquers. Phenol- formaldehyde resins having high degree of polymerization are hard, rigid, scratch-resistant and infusible. They are resistant to non-oxidizing acids, salts and many organic solvents. They can withstand very high temperatures. They act as excellent electrical insulators also. Uses:- They are used for making moulded articles such as radio and TV parts, combs, fountain pen barrels, phonograph records etc. They are used for making electrical goods such as switches, plugs etc. They are used for impregnating fabrics wood and paper. They are used as bonding glue for laminated wooden planks and

	• Sulphonated phenol-formaldehyde resins are use as ion-exchange resins.	
Objective	To prepare phenol formaldehyde resin (Bakelite)	
Procedure	 PFs (Novolac resin) are prepared by reaction of formaldehyde and a molar excess of phenol in the presence of acidic catalyst. The process may be carried out as follows: A mixture of phenol and formaldehyde are allowed to react in the presence of a catalyst. The process involves formation of methylene bridges in <i>ortho, para</i> or <i>both ortho</i> and <i>para</i> positions. These results first the formation of linear polymer (called NOVALAC) and then in to cross-linked polymer called phenol-formaldehyde resin or <i>bakelite</i>. 	
	 Steps: Place 5ml of glacial acetic acid and 2.5 ml of 40% formaldehyde solution in a 500 ml beaker and add 2 grams of phenol. Add few ml of conc. Sulphuric acid into the mixture carefully. Within 5 min a large mass of plastic is formed. The residue obtained is washed several times with distilled water, and filtered product is dried and yield is calculated. 	
	$\begin{array}{c} OH \\ H \\ H \\ \end{array} = O \\ \hline H \\ \hline H \\ \hline C = O \\ \hline H \\ \hline H \\ \hline C = O \\ \hline H \\ \hline H \\ \hline H \\ \hline C = O \\ \hline H \\ \hline $	
	$\begin{array}{c c} OH & OH \\ h & CH_2OH \\ h & + \end{array} n \\ \begin{array}{c} OH \\ \hline \\ OH \\ \hline \\ Polymerisation \\ \hline \\ Polymerisation \\ \hline \\ \\ Novalac \\ (Linear Polymer) \end{array} n$	
	$n \xrightarrow{OH} CH_2OH + n \xrightarrow{OH} Condensation H_2C + C \xrightarrow{H_2} H_2C + H_$	
Observation Table	(Cross-linked Polymer) The weight of the phenol formaldehyde resin isg.	

Determination of	Ring and Ball method (R&B) — ASTM D 3461-76, DIN ISO 4625.		
properties	This method is the most frequently used to determine the softening point of		
	resins. Fig. 1 shows the experimental device used for determination of R&B		
	softening point. The resin is melted into a metal ring and left to cool. The ring		
	is placed in a special metallic device which is placed into a water or glycerol		
	bath. A steel ball of a given diameter and mass is placed on the ring and the		
	bath is heated at a given rate. The temperature at which the ball forces the		
	softening resin downward is noted as the softening point.		
	Thermometer		
	Softening Point		
	Strarting Point End Point		
	Figure 1.Ring and Ball method		
	Novolac resins are soluble in many polar organic solvents (e.g., alcohols,		
Dennender	accione), but not in water.		
Remarks	1. The reaction is sometimes vigorous and it is better to be a few feet away from the beaker while adding the H ₂ SO ₄ and until the reaction is		
	complete.		
	2. The experiment should be preferably carried out in fume cupboard		
Lab Manual	http://ebootathon.com/labs/beta/chemistry/EngineeringChemistryLab/exp1/		
Experimental	https://www.youtube.com/watch?v=mD-5wYpiGRs		
Graphics			
Dolovant IS	https://www.sciencedirect.com/topics/chemistry/solitening-point		
Code			
Quiz	Viva voce:		
	1. What do you understand by resin?		
	2. Describe the uses of the phenol formaldehyde resin.		
	3. What type of co-polymer is phenol formaldehyde resin?		
	4. Briefly explain the properties of phenolic resins.		
	5. write chemical equations for preparation of phenolic resins.		

SEMESTER- 6th

<u>PETROCHEMICALS ENGINEERING</u> <u>LABORATORY</u>

EXPERIMENT-2

Preparation of Base catalysed Phenol formaldehyde resin and Determination of its properties

Introduction	Phenol-formaldehyde resin , also called phenolic resin is made by reacting phenol (an aromatic alcohol derived from benzene) with formaldehyde (a reactive gas derived from methane). Phenol formaldehyde resins (PFs) are condensation polymers and are obtained by condensing phenol with formaldehyde in the presence of an acidic or alkaline catalyst. When PFs are prepared in presence of acidic catalysts are known as Novolacs whereas PFs are synthesized in presence of basic catalysts are called as Resols. Novolacs need a cross-linking agent to fully polymerize whereas resols are self-curing due to the presence of reactive side groups. These are thermosetting polymers.
Theory	A basic (alkaline) catalyst and a molar excess of formaldehyde is used to make resol resins. The following two stages describe a simplified view of the reaction: First, phenol reacts with methylene glycol to form methylol phenol: $HO-CH_2-OH$ Methylene Glycol
	$\begin{array}{c} \overset{OH}{\bigoplus} + \underset{Methylene \ Glycol}{Ho-CH_2-OH} \longrightarrow \underset{H_2O}{\overset{O-CH_2-OH}{\bigoplus} \underset{Methylol Phenol}{\overset{OH}{\longrightarrow} \underset{H_2O}{\overset{OH}{\longrightarrow} \underset{H_2O}{\overset{OH}{\longrightarrow} \underset{H_2O}{\overset{OH}{\longrightarrow} \underset{OH}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{OH}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{OH}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{H_2O}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{H_2O}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{H_2O}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{H_2O}}}}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{H_2O}}}}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{H_2O}}}} \overset{OH}{\overset{OH}{\longrightarrow} \underset{CH_2-OH}{\overset{OH}{\longrightarrow} \underset{H_2O}}}}$
	$\begin{array}{c} & & \downarrow \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \$

1	
	The first part of the reaction, at around 70 °C, forms a thick reddish-brown tacky material, which is rich in hydroxymethyl and benzylic ether groups.
	The rate of the base-catalysed reaction initially increases with pH, and reaches a maximum at about $pH = 10$.
	Being thermosets, hydroxymethyl phenols will crosslink on heating to around 120 °C to form methylene and methyl ether bridges through the elimination of water molecules. At this point the resin is a 3-dimensional network, which is typical of polymerised phenolic resins. The high crosslinking gives this type of phenolic resin its hardness, good thermal stability, and chemical imperviousness
	Because resol resins continue the polymerization reaction at even ambient temperatures, they demonstrate limited shelf lives; dependent on the resin character, storage conditions and application.
	Thermosets: - The polymers which on heating change irreversibly into hard rigid and infusible materials are called thermosetting polymers. These polymers are usually prepared by heating relatively low molecular mass, semi fluid polymers, which becomes infusible and form an insoluble hard mass on heating. The hardening on heating is due to the formation of extensive cross-linking between different polymeric chains. This leads to the formation of a three dimensional network of bonds connecting the polymer chains. Since 3D network structure is rigid and does not soften on heating, the thermosetting polymers cannot be reprocessed.
	 Properties:- Phenol- formaldehyde resins having low degree of polymerization are soft. They possess excellent adhesive properties and are usually used as bonding glue for laminated wooden planks and in varnishes and lacquers. Phenol- formaldehyde resins having high degree of polymerization are hard, rigid, scratch-resistant and infusible.
	They are resistant to non-oxidizing acids, salts and many organic solvents. They can withstand very high temperatures. They act as excellent electrical insulators also.
	 Uses:- They are used for making moulded articles such as radio and TV parts, combs, fountain pen barrels, phonograph records etc. They are used for making decorative laminates, wall coverings etc. They are used for making electrical goods such as switches, plugs etc.
	 They are used for impregnating fabrics wood and paper. They are used as bonding glue for laminated wooden planks and in varnishes and lacquers. Sulphonated phenol-formaldehyde resins are use as ion-exchange
	resins.
Objective	To prepare phenol formaldehyde resin (Resol)

Procedure	PFs (Resols) are prepared by reaction of phenol with formaldehyde in		
	the presence of basic catalyst. The process may be carried out as		
	follows:		
	Steps:		
	4. Place 1 mole phenol and 1.5 mole formaldehyde solution in a		
	beaker.		
	5. Add 5 gm of NaOH in that solution.		
	6. The solution is dried at 80 °C-90 °C temperature for about 1 h.		
	7. A large mass of solidified resil is formed and yield is calculated.		
Observation			
Table	The weight of the phenol formal dehyde resin is		
Determination of	Add 10 ml of solvent (Acetone/ Alcohol/ Petroleum ether/Water) to 0.1 g of		
properties	resin.		
	Write down all the observations.		
Remarks	When the molar ratio of formaldehyde : phenol reaches one, in theory every		
	phenol is linked together via methylene bridges, generating one single		
	molecule, and the system is entirely crosslinked. This is why novolacs (F:P		
	<1) do not narden without the addition of a crosslinking agents, and why		
I ah Manual	Nil		
Experimental Graphics	ame I		
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Experimental Graphics Relevant IS			
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Experimental Graphics Relevant IS Code Quiz	Viva voce:		
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Experimental Graphics Relevant IS Code Quiz	Viva voce: 6. Describe the uses of the phenol formaldehyde resin. 7. Briefly explain the properties of phenolic resins. 8. Evaluation the differences in properties of phenolic resins.		
Experimental Graphics Relevant IS Code Quiz	Viva voce: 6. Describe the uses of the phenol formaldehyde resin. 7. Briefly explain the properties of phenolic resins. 8. Explain the differences in preparation methods between acid catalysed and hase catalysed phenolic resin?		
Experimental Graphics Relevant IS Code Quiz	Viva voce: 6. Describe the uses of the phenol formaldehyde resin. 7. Briefly explain the properties of phenolic resins. 8. Explain the differences in preparation methods between acid catalysed and base catalysed phenolic resin? 9. Write, chemical, equations, for preparation of base catalysed Phenol		
Experimental Graphics Relevant IS Code Quiz	 Viva voce: 6. Describe the uses of the phenol formaldehyde resin. 7. Briefly explain the properties of phenolic resins. 8. Explain the differences in preparation methods between acid catalysed and base catalysed phenolic resin? 9. Write chemical equations for preparation of base catalysed Phenol formaldedyde resin (Resol) 		

SEMESTER- 6th

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EXPERIMENT-3

Determination of Surface tension of Detergent

Introduction	Surface tension is the tendency of liquid surfaces at rest to shrink into the minimum surface area possible.		
Theory	The du Noüy ring method is a technique for measuring the surface tension of a liquid. The method involves slowly lifting a ring, often made of platinum- iridium, from the surface of a liquid. The force, F, required to raise the ring from the liquid's surface is measured and related to the liquid's surface tension, $F = w_{(ring)+}2\pi (r_i + r_a).x$ where r_i is the radius of the inner ring of the liquid film pulled and r_a is the		
	radius of the outer ring of the liquid film. $w_{(ring)}$ is the weight of the ring minus the buoyant force due to the part of the ring below the liquid surface.		
	When the ring's thickness is much smaller than its diameter, this equation can be simplified to: $F = w_{(ring)^+} 4\pi R x$		
	where R is the average of the inner and outer radius of the ring. The measurement is performed with a force tensiometer, which typically uses an electrobalance to measure the excess force caused by the liquid being pulled up and automatically calculates and displays the surface tension corresponding to the force.		
Objective	To determine the surface tension of detergent		
Procedure	 Before the measurement the test body is thoroughly cleaned Anneal it inside the flame of a Bunsen burner Immerse the ring into a detergent solution Repeatedly, pull out the ring from the solution and push the ring into the solution. The tensile force measured reaches extremal values each cycle and the surface tension is calculated. 		
Observation Table	Sl. No.Force (F)Surface tension x123		
Calculation	Surface Tension x =dyne/cm		
Remarks	This method requires that the ring be nearly perfect. Even small blemish or scratch can greatly alter the accuracy of the result.		
Lab Manual	Nil		

Experimental Graphics	
	Figure. Du Noüy surface tensiometer
Relevant IS Code	
Quiz	1. What do you understand by surface tension?
	2. Write the force balance equation of determination of surface tension.
	3. What is the unit of surface tension?

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EXPERIMENT-4

Preparation of Phenol

Introduction	Phenol, also known as carbolic acid, is an organic compound with the chemical formula C_6H_5OH . It is a white, crystalline solid. It has to be handled with care because it causes white blistering to the skin immediately. The crystals are often rather wet and discolored. Phenol contains a phenyl group which is bonded to a hydroxyl (-OH) group. Most batches of commercial liquefied phenol are clear and colourless and can be used in molecular cloning without re-distillation. Before use, phenol must be equilibrated to a pH of >7.8 because the DNA partitions into the organic phase at acid pH.
Theory	 Salicylic acid is decarboxylated by heat to produce phenol and carbon-di-oxide OH OH OH CaO Phenol As the boiling point of phenol and salicylic acid is 181.7 °C and 211 °C, respectively, so phenol is distilled off easily. Uses of Phenol Phenols are widely used as antiseptics (substances that kill microorganisms on living tissue) and as disinfectants (substances intended to kill microorganisms on inanimate objects such as furniture or floors). In addition to acting as an antiseptic, phenol is also a useful precursor in many chemical syntheses to produce pharmaceuticals, food preservatives, polymers, resins and adhesives. Phenolics are also present in a number of biological systems and natural products such as neurotransmitters, flavouring agents, and vitamins.
Objective	To understand the preparation method of phenol

Procedure					
	 Charge boiling Set up regular Since the construction of the	100ml round botto stones. a short path distil distillation apparate he product distilling denser water at ~45 condenser with ho of it. the condensate at to 165°C initially be milky an stillhead temperatur , Collect the pheno coled liquid in the re- g will need to be i tion rate and the wh the residue. he phenol in collect emperature in betwee	om flask with 10 g llation head with us, but you have to s is phenol (m.p. 40 °C. t water to make su the stillhead and nd biphasic from su e will rise and it w ol as a clear, color ecciving flask. ncreased eventuall ole process may tal ting flask again un een 85-90 °C	of salicylic acid and thermometer. You c insulate it really well ^o C) it is prudent to m are none of the pheno heat the distillate b mall quantities of way vill become monopha less to very faintly y y to maintain a reas ce a few hours. der vacuum at 40 mb	l a few an use aaintain ol froze etween ter, but sic dry yellow, sonable oar and
Observation Table	SL. No.	Time	Temperature	weight of the	
		0.51		phenol (g)	
		0.5 h			
		40 mbar)			
		2 h (Vacuum at 40 mbar)			
Calculation	The final yield of phenolg.				
Remarks Lab Manual	 Do not allow the stillhead to exceed 165°C. The residue in the distillation flask is tarry and brown and consists of unreacted salicylic acid, polycondensed crap, and a reasonable amount of phenyl salicylate/xanthone Vacuum is not required necessarily but you will need to do it much higher temperature then. 				
Luy manual			1111		

Experimental	https://www.youtube.com/watch?v=A0e0BDEE4Ic		
Graphics			
Relevant IS			
Code			
Quiz	• What are the uses of Phenol?		
	• Why the temperature of distillation should not cross 165 °C?		
	• What is the colour of distilled pure Phenol?		

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EXPERIMENT- 5

Preparation of Urea formaldehyde resin

Introduction	Urea formaldehyde resin is typically a thermosetting polymer, prepared by the condensation reaction between urea and formaldehyde in neutral or acidic conditions. The synthesis of a urea-formaldehyde resin occurs in two stages. In the first stage, urea is hydroxymethylolated by the addition of formaldehyde to the amino groups. The second stage of urea-formaldehyde resin production includes the condensation of the methylolureas to low molecular weight polymers. The rate at which these condensation reactions occur is very dependent on the pH.
Theory	 Urea-formaldehyde (UF) products (also called aminoplasts or carbamidemethanal) are highly crosslinked, semi-crystalline thermosetting plastics. The UF resins are noted for their high strength, rigidity, cost effectiveness, and fast cure. At elevated temperatures, they can be cured in as little as two seconds. Thermosets:- The polymers which on heating change irreversibly into hard rigid and infusible materials are called thermosetting polymers. These polymers are usually prepared by heating relatively low molecular mass, semi fluid polymers, which becomes infusible and form an insoluble hard mass on heating. The hardening on heating is due to the formation of extensive cross-linking between different polymeric chains. This lead to the formation of a three dimensional network connecting the polymer chains. Since the 3D network structure is rigid and does not soften on heating, the thermosetting polymers can not be reprocessed. Some important examples of thermosetting polymers are Urea-Formaldehyde resin and Melamine- Formaldehyde resins. For the preparation of urea formaldehyde resin, formaldehyde (F) to urea (U) molar ratio should be in between (F/U) 1.8 to 2.5. Properties;- They are clear and white. Better hardness and tensile strength than PF resins. Resistant to most of the solvents and grease. Excellent abrasion resistant and stable to light. Good adhesive characteristics. Good electrical insulators and possess chemical resistance. They are used as adhesives in plywood industries. Uses:- Used for the finishing of cotton textiles (They impart stiffness, crease resistance, fire retardation, water repellency. They are also helpful for shrinkage control)
Objective	To prepare urea formaldehyde resin

Procedure	 Urea and formaldehyde react with each other in neutral or acidic conditions to give mono and dimethylol urea, which undergoes further condensation reaction to give linear, partially cross- linked or fully cross-linked polymer. 1. Place about 5ml of 40% formaldehyde solution in 100 ml beaker. 2. Add about 2.5 g of urea with constant stirring till saturated solution is obtained. 3. Add a few drops of conc. sulphuric acid with constant stirring. 4. A voluminous white solid mass appears in the beaker. 5. Wash the white solid with water and dry it in the folds of filter paper. 6. Calculate the yield of the product 	
	O = C $HCHO$	
	$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	
	Condensation Polymerisation Condensation Polymerisation Condensation Polymerisation Condensation Polymerisation $ \begin{array}{c} \hline \\ \hline $	
Observation Table	Cross-linked Polymer The weight of the urea formaldehyde resin isg.	
Calculation	The yield of urea formaldehyde resin isg.	
Remarks	 Precautions:- Sulphuric acid should be added drop by drop with constant stirring. White solid mass should be removed immediately as it sticks to the walls of the beaker. The reaction mixture should be stirred continuously. The reaction is sometimes vigorous and it is better to be a few feet away. 	

	from the beaker while adding the H ₂ SO ₄ and until the reaction is complete.						
	Safety Instructions						
	H_2SO_4 :- It is poisonous and corrosive. Contact or inhalation can cause severe damage to the eyes, skin and respiratory tract. Wear gloves and dispense under a hood, avoid contact and do not breathe the vapour.						
Lab Manual	http://ebootathon.com/labs/beta/chemistry/EngineeringChemistryLab/exp1/index.html						
Experimental Graphics							
Relevant IS Code							
Quiz	 What do you understand by resin? Urea formaldehyde resin is an addition polymer or condensation polymer. What are the main uses of the urea formaldehyde resin? Briefly describe the properties of Urea formaldehyde resin. Write chemical equations for preparation of Urea formaldehyde resin. 						

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EXPERIMENT-6

To determine the saponification value of a plastic material.

Introduction	Saponification is the hydrolysis of esters. Oils and fats are the fatty acid esters of the trihydroxy alcohol, glycerol. The saponification value of an oil is defined as the number of milligrams of potassium hydroxide required to neutralise the fatty acids resulting from the complete hydrolysis of 1 g of the sample. A soap is formed during saponification, for example: $C_3H_5(C_{17}H_{35}COO)_3 + 3KOH = C_3H_5(OH)_3 + 3C_{17}H_{35}COOK$ Stearin Glycerol Potassium stearate The esters of the fatty acids of lower molecular weight require more alkali for saponification, so the saponification value is inversely proportional to the mean of the molecular weights of the fatty acids in the glycerides present.
Theory	Saponification is the process of breaking down or degrading a neutral fat into glycerol and fatty acids by treating the fat with alkali. It is an index of average molecular weight of the triacylglycerols in the sample. High saponification values of fats and oils are due to the predominantly high proportion of shorter carbon chain lengths of the fatty acids. The smaller the saponification values the longer the average fatty acid chain. If the fatty acids present in the glycerides are low molecular weight (short-chain acids), there will be more glycerides molecules per gram of fat than if the acids are high in molecular weight (long-chain acids). Thus, since each glyceride molecule requires three potassium hydroxide molecular weight correspondingly have higher saponification values. Thus, Saponification values have been reported to be inversely related to the average molecular weight of the fatty acids in the oil fractions. Saponification values are useful in providing information as to the quantity, type of glycerides and mean weight of the acid in a given sample. The saponification value of oils is of interest if the oil is going to be used for industrial purposes. Saponification value is also used in checking adulteration. The larger the saponification number, the better the soap making ability of the oil.
Objective	To determine saponification value of a plastic material

Procedure	I SAMPLE PREPARATION						
	The phenol formaldehyde resin or urea formaldehyde resin can be ta sample.						
	II APPARATUS Bulb condensers Erlenmeyers flasks (50-300 ml depending on sample size) Water bath Pipettes Burette						
	 a) Prepare 0.5N HCl standard solution. Use 1N HCl standard solution and dilute exactly two times. b) Prepare 0.5N Ethanolic potassium hydroxide standard solution. Weigh 35 g of KOH, dissolve in 20 ml of water, then make up to 1000 ml with 95% (v/v) ethanol or absolute alcohol 						
 c) Indicator Phenolpthalein: Take 1 g of phenolpthalein and make up to 100 95% ethanol. Methylene blue: Take 0.1 g of methylene blue and make up t with water. 							
	 IV PROCEDURE 1. Take 2 g of oil in a 50-100 ml Erlenmeyer flask. 2. Add 25 ml of 0.5N ethanolic potassium hydroxide solution an Connect a condenser to the flask. 3. Heat at 80-85°C in a water bath for 2 h. 4. Cool the solution in between 30-40°C and Add 1 ml of indicator titrate with 0.5N HCl standard solution 5. Carry out a blank test (without oil). 						
Observation Table	Sl. No.	Weight of oil	Titration volume of sample	Titration volume of blank test	Saponification value		
Calculation	V CALCULATION Saponification value (mg/g) = (56.1 * (A - B) * F)/ S where 56.1 is equivalent weight of KOH S = sample weight A = titration volume of blank (ml) B = titration volume of sample (ml)						
	F = Normality of HCl standard solution						
Remarks	 Handle HCl solution carefully After completion of saponification process, the flask and the condenser should not be cooled below 30°C otherwise gel will be formed. 						
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Lab Manual	https://core.ac.uk/download/pdf/237468127.pdf						
Experimental Graphics	$\frac{\text{https://www.youtube.com/watch?v=ersHDsiAVko}}{ \bigcirc \begin{tabular}{lllllllllllllllllllllllllllllllllll$						
Relevant IS Code							
Quiz	 Why HCl is used in determination of saponification value of an oil? What is the application of saponification number? How saponification value is determined? 						