

King Fahd University of Petroleum and Minerals

College of Sciences

Chemistry Department



CHEM 399

Summer 2005 (053)

Summer Training Report

## Production of Alkyd Resin

*Prepared for*  
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December 11, 2006

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## **Brief Description of the Company:**

Saleh & Abdulaziz Abahsain Co. Ltd. is among the leading stockiest, traders and manufacturers of chemicals and other products in the Kingdom of Saudi Arabia; it is among the top 40 Saudi Companies holding an impressive profile and is the authorized sole distributor for a number of internationally renowned brands in the Kingdom & other GCC countries.

Abahsain group of companies, whose experience in diversified fields and products has a well recognized and acknowledged name in the industry for the past several years as it is catering a large number of satisfied customers from different backgrounds throughout the Kingdom & GCC.

Abahsain is having a strong presence in market from last 37 years in diversified fields, with its branches all over the Kingdom, UAE and Canada provides a most effective platform for the marketing of various numbers of products. A complete infrastructure covering full service branches located at all major cities; well equipped ware houses, comprehensive material delivery system and trained professionals makes Abahsain a perfect combination to world renowned products distributors. Abahsain has ten active business divisions including Chemical, Machinery Equipment, IT, Pipes/Valves, Automobiles, Food stuffs & Lubricants.

## **Brief Description of the student's work:**

During the summer, I worked in the Chemical Division. Recently Abahsain Chemical Division started a polymeric surface coating resin production facility namely Saleh & Abdulaziz Abahsain Chemical Industry (SABCI). The main products of SABCI are Alkyd Resin, Acrylic Emulsions, Unsaturated Polyester resins and PVA Resins. The plant started operation in the middle of June 2006. Estimated production capacities of Alkyd Resins and Acrylic Emulsions are 2600 metric tons per year and 2300 metric tons per year respectively. PVA resins trial production is under progress while the license for production of unsaturated polyester resin is pending. Most of my work was with the production department which is responsible for the production of the alkyd resins.

## **Introduction:**

Alkyd resins form one of the biggest, if not the biggest, genre of surface coatings resins. They date from the late twenties as commercial products for paints and their use has encompassed all the surface coatings market segments, from automotive to decorative, from marine to aerospace.

The name alkyd was proposed by Kienle in 1927 and is the euphonic contraction of 'al' from alcohol and 'cid' from acid, (rewritten as 'kyd' to aid pronunciation).

Alkyds are polyesters containing a fatty acid chain and are the product of the reaction of polyhydric alcohols, polybasic acids and fatty monobasic acids. The history of the development involves the first polyesters prepared by Berzelius in 1847, the result of the reaction between glycerol and tartaric acid. Through the remainder of the nineteenth century, various polyesters were prepared, all using glycerol and varying the acid: camphoric, succinic, citric and phthalic. However, it wasn't until the General Electric Company patents of 1910 for the use polymeric esters as varnishes, impregnating resins and adhesives that any use for the materials, by now modified with monobasic acids such as butyric or oleic, was found.

In 1914, Howell made glycerol phthalate modified with castor oil and this is the recorded preparation of what was later to be called an alkyd.

In 1921, Kienle showed that vegetable oils could be incorporated into the polyesters by the alcoholysis technique, rather than use the fatty acids. In 1927, the monoglyceride process was discovered by Ott, et al for the preparation of alkyds.

Progress to commercialization for alkyd resins was slow in these early years due to the high cost of phthalic anhydride. In 1916, the Gibbs-Conover process for the manufacture of phthalic anhydride from naphthalene was introduced and for the first time phthalic anhydride became plentiful and moderately priced. Phthalic anhydride moved

form \$ 4.23 per pound in 1917 to 16 cents a pound in 1928. In 1928 around a half million pounds of alkyd resin was produced in the USA.

This was to be far from the last time that alkyd technology drove raw material innovation and manufacture, pentaerythritol was commercially produced from formaldehyde and acetaldehyde in 1937. Dehydrated castor oil was produced in quantity during the Second World War due to the shortages of tung oil. Immediately post war, the trimethylols of propane and ethane were available. In 1947 glycerol was produced from petroleum rather than fats and oils and the price of this key raw material was reduced and stabilized. By 1956, isophthalic acid was commercially available.

The first alkyd resins sold commercially were marketed as "Glyptals" by the General Electric Company. Initially, blending of soluble glycerol phthalate resins with the drying oil was not possible, due to their incompatibility. It was not until fatty acid were introduced into the reaction of glycerol and phthalic anhydride that a homogenous resin could be produced which would dry by oxidation, i.e. become insoluble after film formation.

In the 20's nitrocellulose lacquers were introduced into the embryonic automotive industry to speed throughput due to their shorter drying times. However, the adhesion of the nitrocellulose lacquers to the substrates was poor and it was discovered that compatible rosin modified alkyds and non drying oil alkyds could improve the adhesion of the nitrocellulose lacquers, and the alkyd industry really began to take off.

Alkyds were now ready for use as reasonably priced resins for the industrial coating markets and their huge potential began to be realized. They were modified with phenolic resins, reacted with styrene, silicone and epoxies. They were used with amino resins to produce the first stoving finishes and found end uses as diverse as automotive, domestic appliance, architectural, marine and aircraft. They were the coating technology of choice for almost all applications until the late fifties, early sixties when latex paints began to make inroads in the architectural markets and acrylics in the domestic appliance

market and later in the automotive and aerospace markets. Later still, the advent of powder coatings further eroded the presence of alkyd technology in the paint markets.

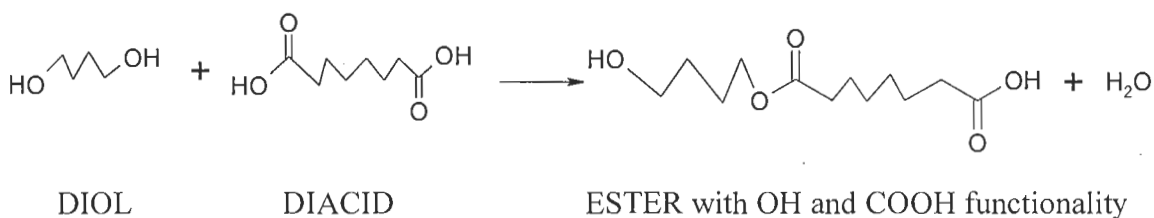
However, alkyds remain a powerful technical solution to paint formulators, they have, from way back, had waterborne applications as solution polymers in water and more recently as alkyd emulsions. They lend themselves to high solids formulations and their ability to form stable (in the can) one – pack ambient cure formulations ensures that the technology has not run its course yet!

## **I. General Preparation of Alkyds:**

Alkyds are classified as step growth or polycondensation polymers. A condensation polymer is one where the molecular structure of the repeat unit lacks certain atoms which were present in the monomers from which it was prepared.

Alkyds are prepared either by the direct esterification of carboxylic acids with alcoholic hydroxyl groups, transesterification or reaction between glycols and other polyols with anhydrides.

### **A. Direct Esterification:**



This reaction is either self catalyzed by a second molecule of carboxylic acid or by an independent acidic catalyst.

Following mechanism is proposed for self catalyzed polymerization:

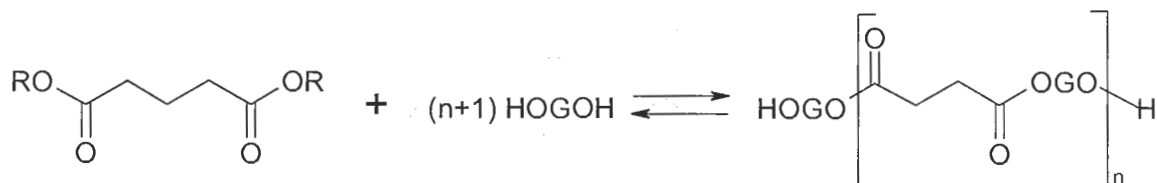


The oxonium ion arises from the autoprotolysis of the acid and the above is the rate determining step.

By analogy, the catalyzed polymerization mechanism has second order kinetics so that a typical catalyst would be para-toluene sulphonic acid.

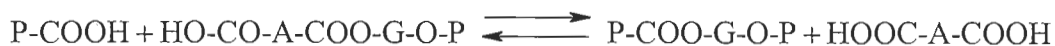
### **B. Transesterification:**

Transesterification can occur by alcoholysis or acidolysis. In the alcoholysis route, transesterification can be considered to take place in two steps, in the first the diester and a glycol react, with excess glycol driving the reaction:



The second step is a polycondensation at higher temperatures with the elimination of glycol.

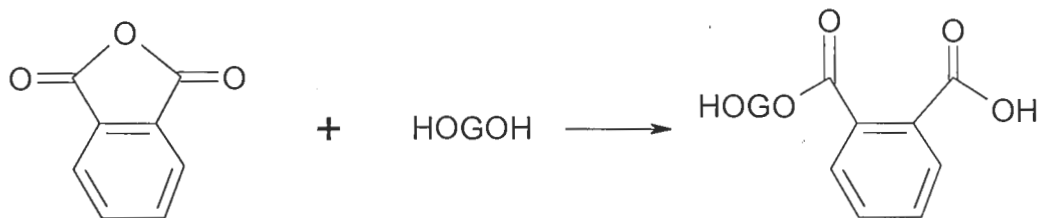
In the acidolysis route, a carboxylic acid is reacted with an acetate ester:

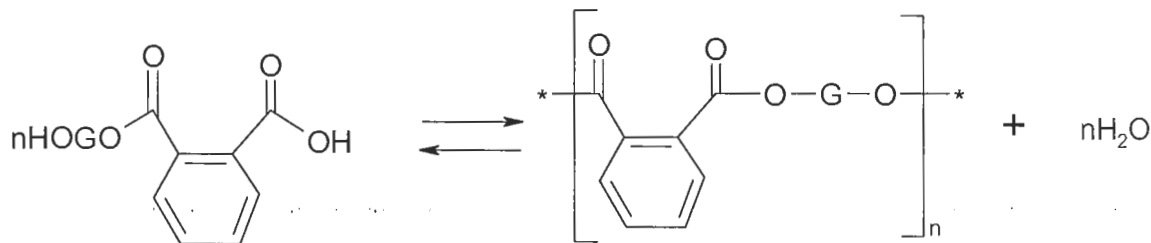


Acidolysis is similar to alcoholysis but is much less important commercially.

### **Reaction between polyols and anhydrides:**

This is the most important commercial route to alkyds and polyesters. The first step is ring opening of the anhydride followed by the rate determining esterification step:





In the plant I worked in, we prepared alkyd resins by transesterification by alcoholysis. First the transesterification takes place by reacting triglycerides from oil polyols and then the acid was added for polycondensation reaction to take place. Details are given under the heading Manufacture of Alkyd Resins.

## II. Classification of alkyd resins:

Alkyds have been classified by 'oil length' and it is a convenient if somewhat inaccurate method of dividing them into three broad groups. It is inaccurate in the term that oil length is valid only if the alkyd is glycerol as the oil length is defined as the percentage fatty acid in an alkyd expressed as triglyceride.

The following table illustrates the convention.

### Classification of Alkyds

Classification	% Fatty Acid (as triglyceride)	Phthalic Anhydride %
Short Oil	32-40	38-46
Medium Oil	43-54	30-37
Long Oil	55-68	20-30
Very Long Oil	>68	<20

Before going into details of the process, I would like to discuss about the raw materials used and their specifications:

### **III. Raw Materials:**

#### **A. Oils:**

Oils are used as raw materials in alkyds to impart flexibility, to allow ambient temperature cross linking through oxidation of the fatty acid double bonds and to allow/promote flow, wetting and leveling during film formation.

Alkyds are made with drying, semidrying and non-drying oils or their fatty acids. This classification into drying, semi-drying and non-drying was carried out by Jamieson in 1954 according to iodine number.

Iodine number or iodine value of oil is a convenient measure of the double bond content of oil, which in turn is a reflection of the oxidative drying capability of the oil. The method gives an accurate measure of the number of double bonds present where the double bonds are not conjugated. It was proposed that oils with iodine number greater than 140 were drying oils, Iodine numbers approximately 125-140 were semi-drying and below 125 were non-drying.

A further important term used with regards to oils is Saponification Value. When a glyceride is reacted with an alkali it is saponified or decomposed into the alkali salts of the fatty acids and glycerol. The saponification (or sap) value is defined as the number of milligrams of potassium hydroxide required to saponify 1 gram of oil. Drying oils contains small quantities other than esters and these are not saponified on boiling with alkali. One percent is normal maximum and figures above this denote levels of impurity.

The oil which we used is soya bean oil. Although other oils like sunflower oil, Castor oil, crude fish oil etc can also be used.

## **1. Soya Bean Oil:**

Soya bean oil is produced from the beans of the plant *Glycine max*, which is grown in temperature regions of the USA, Brazil, Argentina and China.

Crude soya bean oil is extracted from soya beans by solvent extracting, with a yield of 15-18% and it is the largest volume vegetable oil produced in the world with a high proportion refined for edible purposes. Soya bean oil is thus a major raw material and it is processed to give a range of products for technical applications.

Raw or crude oil produced contain variable amount of non-glyceride impurities. For alkyd resin manufacture, these impurities are undesirable and need to be removed by refining.

The principle impurities are free fatty acids which may account for several percent by weight of oil. Other impurities include phospholipids sterols, tocopherols, hydrocarbons, pigments, sterol glucosides and protein fragments in varying minor amounts.

Treatment of the crude oils with phosphoric acid prior to neutralization helps with the removal of the phosphatides. Free fatty acids are removed by neutralization with caustic soda to produce soaps, which are centrifugally removed, along with the break, and the remaining oil is then water washed.

After drying, the partly refined oils are then bleached with activated earth, which absorbs color bodies to give oils which are clear and bright, pale in color, low in acid value and generally free of any form of precipitates. They are known as alkali refined oils.

The specifications of the oil we used is given on the following page:

Parameters*	Unit	Value**
Free Fatty Acid	%	0.028
Color (5.52" Cell)	R / Y	0.5 / 5.0
Peroxide Value	m eq. / Kg	0.65
Iodine Value	%	131.4
Rancidity	-	Negative
Taste	-	Bland
Odor	-	Neutral
Moisture	%	0.012
Clarity	-	Clear
MOT	-	Negative
Soap Test	-	Negative

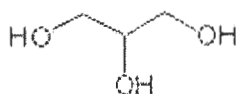
\* AOCS method of analysis

\*\* Analysis at the time of filling/ packing/ bulk loading

## B. Polyols:

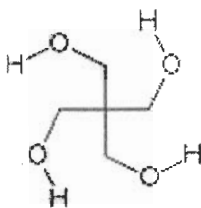
Many diverse chemical compounds are used as polyalcohols in the preparation of alkyd resins. The information below relates to those polyols only which were being used in the plant.

### 1. Glycerol:



Glycerol, a triol, is the first polyol to be used in the production of alkyd resins. It contains two primary hydroxy and one secondary hydroxy group which all react with the same rate with fatty acids to form a triglyceride. With dibasic acids the primary groups react more readily than the secondary; and in an alkyd formulation, it is likely that the former react with phthalic anhydride leaving the secondary to react with the fatty acid. Glycerol is used in alkyd resins of all oil lengths.

## 2. Pentaerythritol:



Pentaerythritol (Penta) is a solid with a melting point of 253°C and is manufactured by reaction between acetaldehyde and formaldehyde in the presence of water and alkali. It contains four primary hydroxyl groups and forms more complex resins with phthalic anhydride than does glycerol.

Penta is used principally in long and medium/long oil alkyd resins, as the long and middle oil lengths make it possible to take advantage of pentaerythritol's properties. A medium to short oil alkyd based solely on pentaerythritol would be prone to gel too rapidly.

If pentaerythritol is mixed with dihydric alcohol, it may be used with short oils. Mixtures of pentaerythritol and ethylene glycol are frequently used to produce medium and short oil alkyds containing 30-50% fatty acids. These resins have been shown to exhibit better compatibility, gloss retention and durability properties than the alkyds based solely on glycerol. However, the volatility of ethylene glycol presents process problems.

Long oil alkyd paints made with pentaerythritol have superior adhesion, weather-resistance, color, lustre, water resistance and chemical resistance properties in comparison with trihydric alcohols, such as glycerol, and then faster than trihydric alcohols.

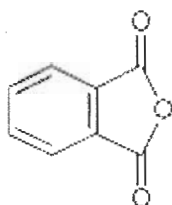
Pentaerythritol can be used to produce synthetic drying oils of good quality by esterification reaction with linseed oil, tung oil, or other natural drying oils, or by ester exchange and other reactions with fatty acid esters. These synthetic drying oils dry quickly, and because they have outstanding properties,

such as lustre, water-resistance, alkali-resistance and flexibility, they are widely used in producing printing inks and processing paper.

The above polyols are the most widely used, mostly because of low cost and wide availability, but other polyols are used, but either in small amount or for a very specific applications.

## **C. Polyacids:**

### **1. Phthalic Anhydride:**



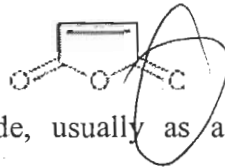
Orthophthalic acid, used only as its anhydride derivative, remains the most employed diacid in the alkyd resin industry. Originally, phthalic anhydride was produced solely from the oxidation of coal-tar-derived naphthalene and its availability depended on the availability of coal tar from coking operations. In 1960, when coal tar naphthalene was in short supply, output was erratic and prices fluctuated. As a result, petroleum-derived naphthalene was commercialized as a new feed stock for phthalic anhydride. However, naphthalene supplies remained unstable, causing many producers to start using O-xylene as a raw material. Since that time, the petrochemical industry has rapidly expanded, making large quantities of o-xylene available at competitive prices. Consequently, o-xylene has become the predominant starting material for the production of phthalic anhydride.

The largest markets for phthalic anhydride, plasticizers, unsaturated polyester resins and alkyd resins are all relatively mature and therefore subject to the influence of general economic conditions. Phthalic anhydride gives alkyd

resins with hardness and chemical resistance, due to the phenyl structures resistance to rotation. This structure also causes alkyds made from phthalic anhydride (acid) to be susceptible to hydrolysis.

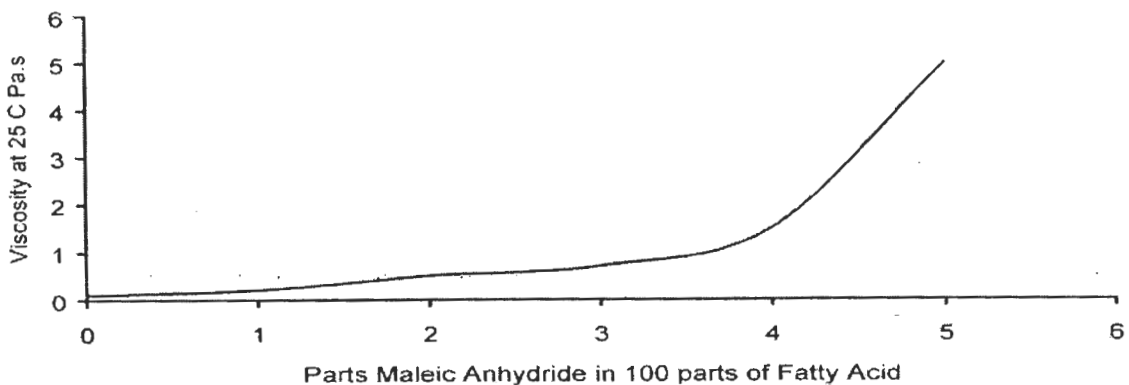
In many ways, the choice of phthalic acid and a fatty acid defines all that is both good and bad about alkyds. Phthalic is the acid of choice because of cost, fatty acids are used to enable ambient cure, ease of surface wetting and cheapness. This sums up alkyds, cheap, effective, and easy to use. Down sides are poor exterior durability and yellowing – both of these attributes can be avoided by not using phthalic acid and not using fatty acids, but that puts both the economics and the ease of use out of the window. i.e. it throws the baby out with the bath water!

## 2. Maleic Anhydride (MA):



Use of this anhydride, usually as a partial replacement of phthalic anhydride, allows notable improvements in the drying properties and hardness of the resin. It is employed in the maleinisation of fatty acids which can lead to water dilutable alkyds. It is often used effectively to increase the viscosity of a resin which will then require a large volume of dilution solvent thus reducing the cost of the alkyd. Its use is normally restricted to between 1 and 10% replacement of the phthalic anhydride content and the figure below shows the influence of MA on the viscosity of a long oil soya alkyd.

### Effect of Maleic Content on Viscosity of Long Oil Soya Alkyd



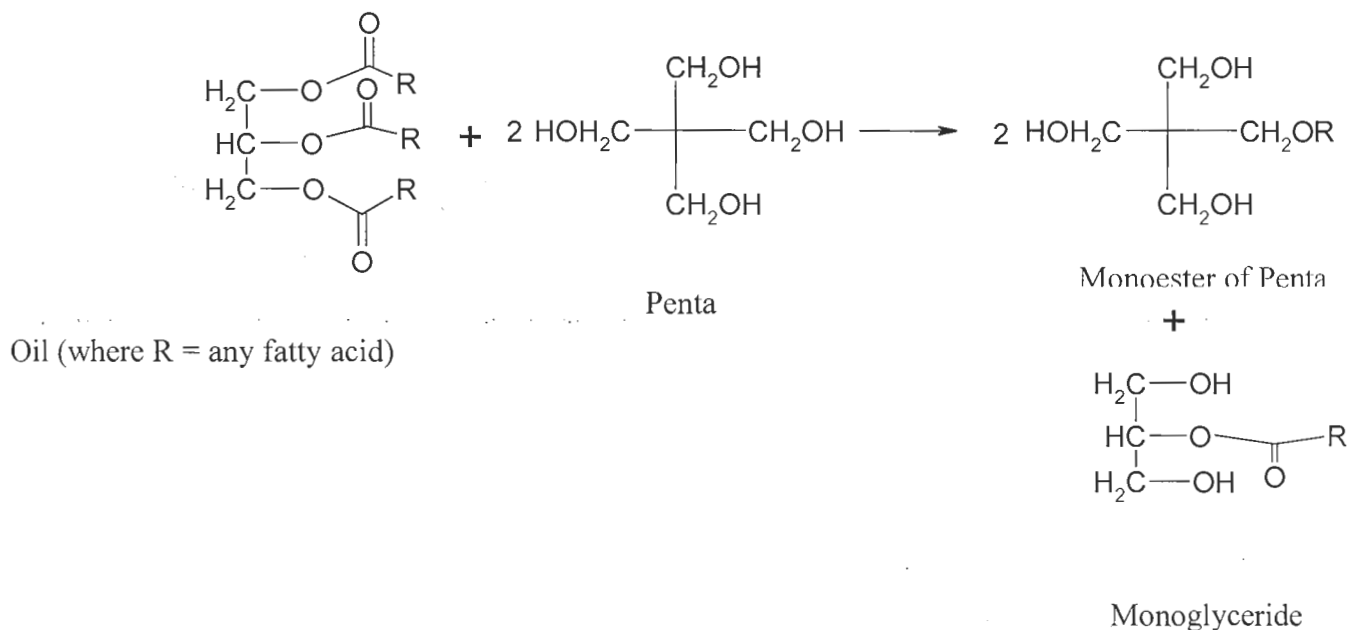
#### IV. Manufacture of Alkyd Resins:

##### Alcoholysis:

In a manufacture of alkyd resins, alcoholysis was the first reaction carried out. The soya bean oil is reacted with pentaerythritol. Due to ester interchange the fatty acids arrange themselves among the excess of hydroxyl groups; the goal is to convert the total charge to monoester; the phthalic anhydride was then added and the resin is processed to completion. Since the phthalic acid also requires hydroxyl group with which to react, the alcoholysis was undertaken in the following way:

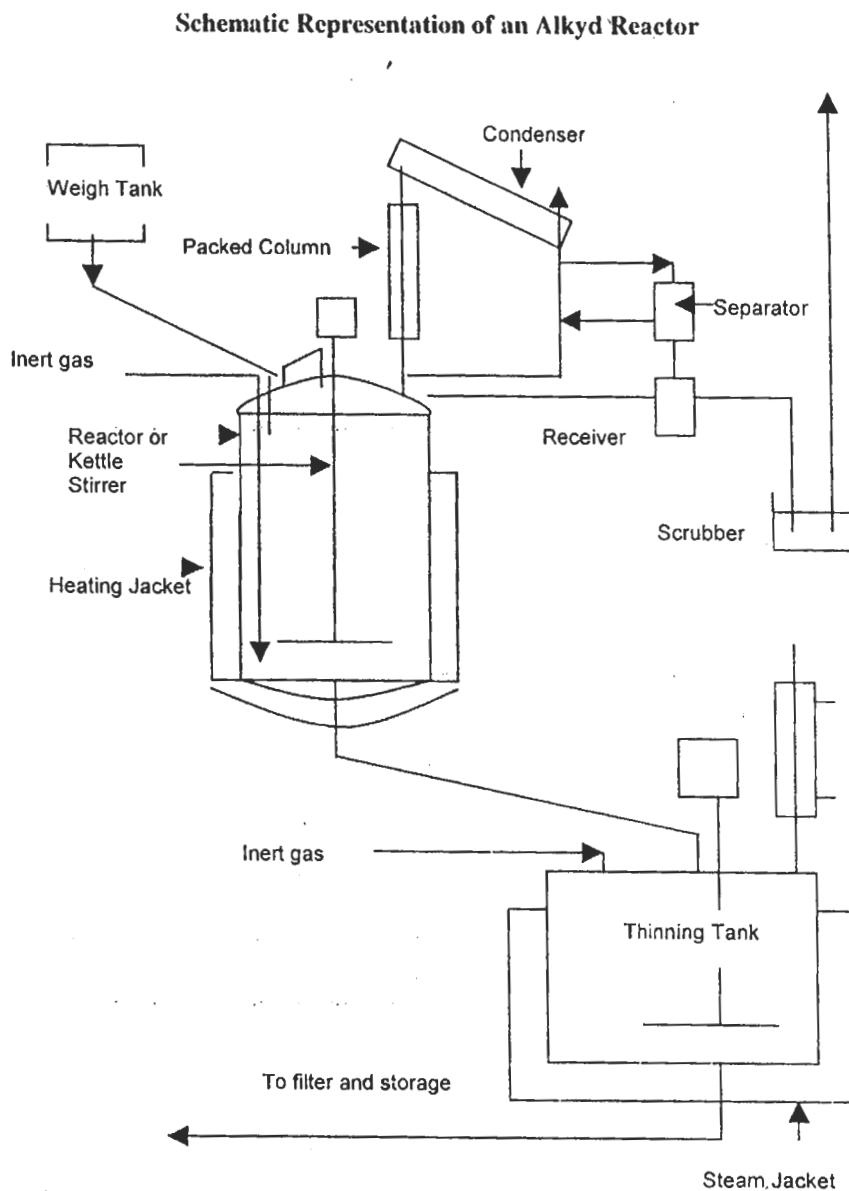
- By preparation of a theoretical monoester with one mole of oil and two moles of polyol and subsequent addition of extra polyol at the same time as addition of polybasic acid

Usually, molar ratios higher than 1:2 were used, giving an excess of hydroxyl groups in the alcoholysis product.



## Reactor:

Systematic diagram of the reactor and other utilities is shown below:



## Catalysts:

The alcoholysis reaction proceeds very slowly below  $200^{\circ}\text{C}$  and it is necessary to use temperature of  $280^{\circ}\text{C}$  or higher to achieve reasonable reaction rates. Considerable darkening of the components can take place at these high temperatures. Fortunately, the

reaction can be fairly readily catalyzed, enabling the alcoholysis to be carried out at about 220°C in relatively short times.

The catalyst used was a mixture of lithium Hydroxide and lead octoate.

After completion of the alcoholysis and subsequent esterification of the alkyd, the catalyst is converted to the metal salts of the dibasic acid used. This salt was insoluble and was removed during the final alkyd filtration.

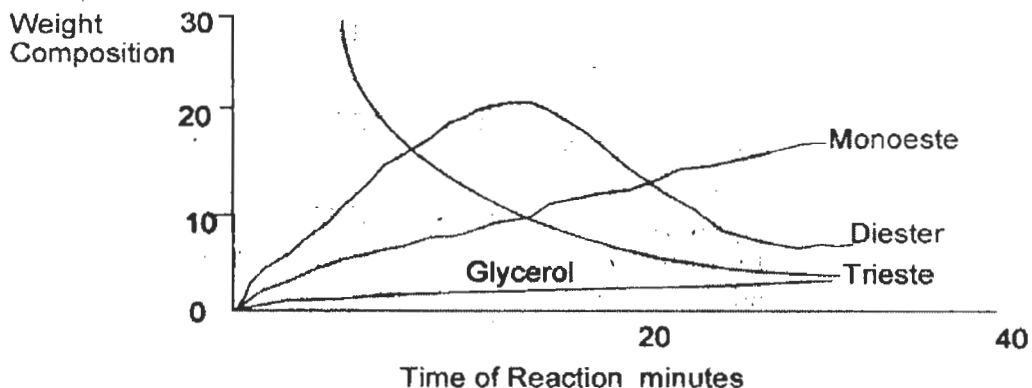
### **Control of Alcoholysis:**

When the oil and pentaerythritol are first mixed, they are incompatible, and very little reaction ensues even at high temperature. However, as soon as a small amount of monoester has been formed, the combination of all three components is compatible and the reaction gathers pace. Since there are no acid or hydroxyl value changes and no water is evolved, there are no simple chemical tests for estimating monoester content and physical methods were used.

The extent of alcoholysis was measured by alcohol solubility since the monoester formed is soluble in alcohol and the oil is insoluble. The test is called Methanol Tolerance Test (MTT). A solubility of 1 to 3 by volume with methanol is considered to be an indication of sufficient monoester formation to proceed with esterification. This solubility corresponds to about 40% monoester formation. Sometimes, when the solubility was in doubt, due perhaps to insoluble pentaerythritol, a practical test was done. Some of the alcoholic mixture was heated with phthalic anhydride (or other acid being used) in the proportions to be used in the batch. If satisfactory, the mixture will become homogeneous, if not; a grainy, insoluble layer will separate.

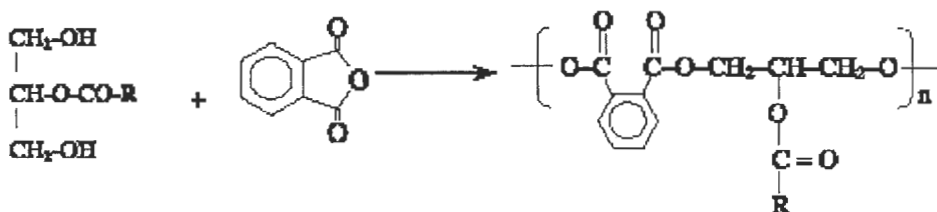
The scheme on the following page shows the change in composition in a monoglyceride process:

## Alcoholysis of Oils



### Esterification:

The esterification reaction began as soon as phthalic anhydride along with the remaining pentaerythritol is added to the reactor. Some times maleic anhydride was added to replace some of the phthalic anhydride for the reasons mentioned before. The general reaction that takes place is as follows



The reaction of monoester of penta and phthalic acid is much more complex and will not be shown here.

The esterification stage of alkyd resin manufacture is a condensation reaction; water is evolved as a by-product. Since the esterification is an equilibrium reaction, the evolved water must be removed to force the reaction to completion. The technique used to remove water is called the solvent process.

### **Solvent Process:**

The esterification reaction was carried out in the presence of a xylene (solvent), which is miscible with the reaction bulk and immiscible in water when cooled. The boiling point of xylene, is lower than the polycondensation temperature. Water given off by the reaction is carried over as an azeotrope with xylene, which then condenses into a separation funnel where the two products separate out. The water is drawn off and the xylene is returned to the reaction vessel.

Reactant losses are limited because the whole reaction vessel is swept by the xylene vapors, which wash the reaction vessel walls and recycle the products that have sublimed and crystallized.

### **Control of Esterification:**

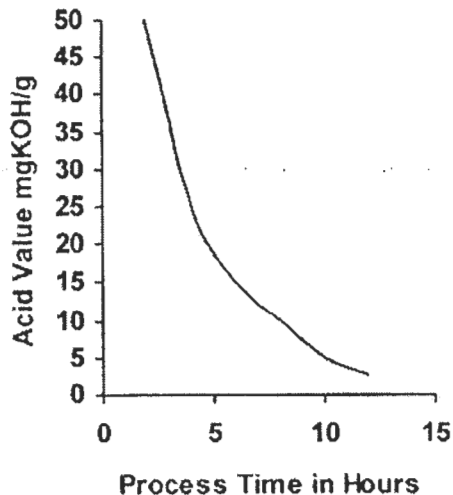
The esterification was controlled by viscosity increase and acid value decrease. Samples were taken from the batch at pre-determined intervals after processing temperature has been reached.

The samples for viscosity measurement were diluted with solvent, which was usually the same solvent and in the same ratio that the batch is to be finally manufactured. Acid value was determined by titrating with alcoholic potassium hydroxide solution. (Details follow)

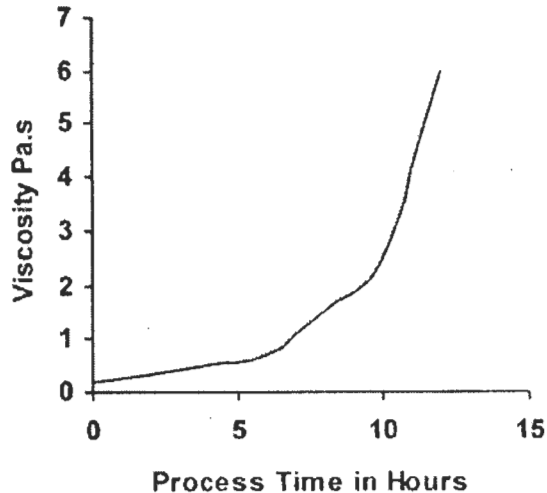
In processing it must be remembered that the viscosity and acid value determination takes approximately twenty minutes to carry out, with the batch continuing to change during that time. When processing by the solvent method, the sample taken from the reaction will contain a percentage of reflux solvent and this has to be allowed for while process testing. Typical graphs of acid value and viscosity are shown on the following page:

## Acid Value/Viscosity Graphs

Acid Value against Process Time



Viscosity increase with Process Time



At a pre-determined viscosity and acid value, the batch is cooled and the product is transferred in a dilution tanks where the product are adjusted according to the requirement. Filtration is then carried out and the final product is packed in drums.

### Tests for Esterification Control:

Following two tests were conducted for each sample drawn out during the process:

- Viscosity
  - i. By Ford Cup
  - ii. By Cone & Plate Viscometer
- Acid Value

## Viscosity:

The viscosity is usually measured by Ford Cup method. Cone Plate is also used if the alkyd formulation gives viscosity values in poises. The two methods and procedures are given on the next page:

### Viscosity by Ford Cup:

Measuring the viscosity by Ford Cup is a standard method of measuring viscosities of alkyd resins in the industry. The viscosity is measured in seconds. The basic idea is to measure it takes to empty a ford cup full of resin. The figure of the ford cup used is shown below



It is important to mention the temperature and method number with the results. We use to measure the viscosity at 25°C using ASTM D 1200. Usually the resin is thinned to 50% using the thinner to be used in the final product. The standard procedure is given below:

### APPARATUS:

1. Viscometer
2. Thermometer
3. Stop watch

### **REAGENTS:**

1. Ice cold water

### **PROCEDURE:**

1. Make sure the ford cup is clean and dry.
2. Cool the resin to 25°C by dipping the sample container in ice cold water.
3. Fix the ford cup in the stand and place a beaker under the ford cup.
4. Close the nozzle of the ford cup and fill it with the resin up to the mark.
5. Remove the finger from the nozzle and turn on the stop watch simultaneously.
6. Read the time when the sample is completely drained.
7. Report the time in seconds.

### **PRECAUTIONS:**

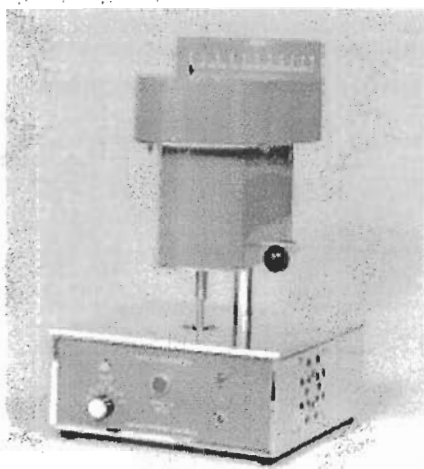
1. The ford cup; especially the nozzle, should be completely clean.
2. The viscosity of the resin depends strongly on the temperature. The temperature should be maintained at 25°C as much as possible.

### **NOTE:**

Make 50% solution of alkyd resin in the required thinner for measuring viscosity by Ford Cup

## **Viscosity by Cone & Plate Viscometer:**

Cone & Plate Viscometer is not used very often for measuring the viscosities. It is usually used when the viscosity values in the alkyd formulation is given in poises. The advantage of Cone & Plate Viscometer over Ford Cup is that the viscosity is measured at 150°C which is automatically maintained. So temperature is constant during the process, thus giving accurate values. Also it takes much less time as there is no need for cooling the resin to room temperature, the viscosity is measured for pure resin (without thinning) and lastly, it consumes very little amount of sample. The figure of the apparatus is given on the following page:



The general experimental procedure follows:

### **APPARATUS:**

1. Cone and Plate Viscometer
2. Thermometer

### **REAGENTS:**

1. Ice Cold Water

## **PROCEDURE:**

1. Turn On the viscometer at least 10 minutes prior to testing and set the required temperature (150°C).
2. Cool the resin to a temperature little less than 150°C by dipping the sample container in ice cold water.
3. Put the resin sample in the sample holder and wait for the temperature to stabilize (Indicator light turns green when the sample temperature is stabilized at the set point)
4. Push the read button to read the viscosity in poise.

## **NOTE:**

No sample dilution should be done.

The sample holder should be thoroughly cleaned with a solvent.

## **Acid Value:**

The acid value of the resin is simply a measure of free acid content of the acid. The acid value should be controlled around 10 mg KOH per grams of resin. Higher acid value causes problems after the film formation. They cause inferior coatings because of moisture sensitivity imparted by the unconverted COOH groups in the films. On the other hand low acid value requires more reaction time; thus higher energy expenditure. Longer reaction times results in darker color due to prolong cooking and also very high viscosity products, which eventually becomes difficult to handle.

The experimental procedure for calculating the acid value is given below:

## **APPARATUS:**

1. Conical flask 250 ml,

2. Burette
3. Analytical Balance

### **REAGENTS:**

1. 0.5 N Potassium hydroxide
2. Ethyl Alcohol (Purity 99.5 %)
3. Phenolphthalein

### **PROCEDURE:**

1. Weigh approximately about 2 gm of sample into 250 ml conical flask and add 30 ml of alcohol.
2. Add a drop of indicator and titrate the solution with 0.5N KOH solution while shaking until the pink color just appears which will persist for 30 seconds.

### **CALCULATION:**

$$\text{The acid value} = \frac{\text{Volume of KOH} \times \text{Normality of KOH} \times 56.1}{\text{Weight of Sample}}$$

### **V. Corrective Measures:**

High acid values during processing were reduced by glycerol additions even in pentaerythritol based alkyds. It is better to use glycerol since it reacts fast and is more readily soluble. If the acid value is lower than expected, small amount of phthalic anhydride was added during processing.

Slow-bodying (bodying is term used for viscosity increase) were processed at higher temperatures with flow of inert gas, CO<sub>2</sub>. Exclusively fast bodying batches were processed at lower temperature until they become controllable.

Batches that finished exclusively high in viscosity were reduced in viscosity by adding more oil to the reactor. Once the viscosity is lowered, additional amount of phthalic anhydride and pentaerythritol was added to re-process into specification.

## **VI. Sample Batch Report:**

One of the batch report done during the summer training shown in the following page.

**S & A ABAHSAIN CHEMICAL MANUFACTURING INDUSTRY**  
**COOKING LOG SHEET**

PRODUCT 6070

Charging Date 20-07-06

Batch No. 20-07-06-001 Plant No. CNE

Filling Date \_\_\_\_\_

TIME	TEMPERATURE		REACTOR COLUMN		VISC	Acid Value	COLOUR	I.G. FLOW	REMARKS
	SET	PLANT	INLET	OUTLET					
Hours	°C	°C	°C	°C	Poises			m <sup>3</sup>	
1215	140c								Feed SBO
1225	140c		44c						Heating ON
1245	140c		43c						Added c2321, L. Hydroxy
1255									
1355	165c		125c						
1455	175c		160c						
1555	195c		180c						
1518	210c		190c						
1555	230c		200c						
1630	242c		205c						Octate
1700	255c		220c						Added Lead Octate
1730	260c		235c						
1800	270c		240c						
1815	270c		245c						M.T.T Not clear
1845	270c		252c		Heating OFF				M.T.T Semi clear
1910			245c						M.T.T clear
1945			240c						P.A test OK
2010	Dnc to cooling pump problem								Cooking started
2000									Added c2610, c275
2030			155c						Added xylene 250g
2035	195		155c						Heating on
0500	253		225c		1.05	31.4	32 F/5c		
0600	235		221c		1.7	25.4	38 F/5c		Added xylene 100g for Reflux
0700	235		218c		2.1	21.9	55 F/5c		
0745	220c		215		2.4	21.0	58 F/5c		
0810									Added Calc 58.4g
0810	220		211						xylene 50
0840	220		209		2.4	21.0	55 F/5c		
0915	225		205		2.3	21.0	50 F/25c		
1010	225		211		2.6	19.6	SMTMT 50 F/5		
1050	220		211		3.0		50 F/5c		TOTAL WEIGHT:
1120	Heating OFF		211		3.4	17.0	50 F/5c		FINAL VISCOSITY:
1145									FINAL ACID VALUE:
1220			172						REMARKS:
1246									Cooking started
1317									Cooking stopped
									By Transfer into T.V
									Complete Transferring

## **VII. Final Testing of Alkyd Resins:**

Number of tests are conducted before the product is handed over to the quality control department. These tests are as follows

- Final viscosity
- Acid Value
- Solid Content
- Color

### **A. Final Viscosity:**

The final viscosity of the resin is measured by Ford Cup. (Details of method and procedure are given before)

### **B. Acid Value:**

Details of method and procedures are given before

### **C. Solid Content:**

The purpose of checking solid content of the finished product is to make sure that the dilution is accurately done. The basic idea of the test is to evaporate a pre-weighed thinned resin and heat it at the temperature at which the solvent evaporates and the non-volatile alkyd resin remains. As we were using white spirit (boiling temp.  $<150^{\circ}\text{C}$ ) we used to set the temperature of oven at  $150^{\circ}\text{C}$ . General experimental procedure is given below:

### **APPARATUS:**

1. Analytical balance
2. Watch glass

3. Spatula
4. Electrical Oven.

### **PROCEDURE:**

1. Wash the watch glass with distilled water and keep in oven for drying.
2. Weigh the empty Watch Glass.(W1)
3. Weigh about 1 gm of sample in watch glass.(W2)
4. Keep the sample in oven at 150°C for 15 minutes.
5. Remove the sample and allow it to cool at room temperature.
6. Weigh the dried sample.(W3)

### **CALCULATION:**

$$\text{SOLID CONTENT \%} = \frac{W_2 - W_1}{W_3 - W_1} \times 100\%$$

### **D. Color:**

The color of the resin is important to control as it affects the color of paint in which it is used especially if the color of the paint is light. The color of the resin is measure by a color comparator. A typical apparatus is shown in the figure below.

The apparatus has a film of standard colors in which the lightest color is given the value of 1 and the darkest being 9. The color of the resin is compared with the standards and thus given a value from 1 to 9 depending where the range of the color of the product lies. If the color of the resin lies between two digits, lets say between 3 and 4, the value is reported as 3+. The resin used for color testing is thinned to 50%.

### **Conclusion:**

I completed 8 weeks of training in SABCI. I spent my time in working in the production department in alkyd resin manufacturing unit. I worked in the team responsible for alkyd resin manufacture and product testing. I feel that a good hand-on experience is gained by working in this area. I would like to thank SABCI for offering this opportunity to work with them and gain such a great experience.

## **References**

1. N Tuck. Waterborne and Solvent Based Alkyds and their End User Applications Volume VI. USA: John Wiley & Sons Ltd, 2000
2. Oil and Colour Chemists' Association. Surface Coatings Volume I. USA: Chapman and Hall, 1983