

## Enamel paint based on short oil alkyd

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**Abstract:** The present research program proposed to prepare short oil alkyd with a very low oil length 7 to 15 %. Normally it is difficult to cook short oil alkyd with such low oil content however we have attempted the control of reaction by using chain stoppers like rosin and benzoic acid. The main idea is to control the reaction by using appropriate amount of chain stoppers rosin and benzoic acid.

Short oil alkyd based on short oil with very low oil length i.e. 7 to 15 % has been prepared. Looking to the practical problem of high temperature reaction 275 to 280 °C, low temperature reaction 220 to 200 °C have been tried to get better performance and control of the reaction. These short oil alkyd compositions have been converted to alkyd emulsion paste containing 30 to 56 % water. Alkyd emulsion paste with low acid value and high % solid (50%) were to be prepared. These alkyd emulsion pastes have been used in formulation of synthetic emulsion paint. The proportion of emulsion paste binder is 15 to 30 % to get the desirable characteristics of paints. The emulsion paints is compared against commercial paint for the view point of technical and economical viability.

**Keywords:** Short oil alkyd, Novel resin, synthetic emulsion paint

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### 1. INTRODUCTION

The surface coating industry is always in search of a water thinnable coating medium, which will have excellent hardness adhesion and resistance characteristics. Our institute is working on paint binder based on castor oil. We have developed several paint binders such as Castro alkyds and sorbo alkyds. At presents the acrylic binder are fast replacing oil base binders. The main disadvantage of oil binder is a sluggish drying property.

### 2. Experimental Details

#### a) Materials

A short oil alkyd resin<sup>1</sup> were prepared, raw material required, grade of material, source of material and specification have been given in table no.1. Alkyd resins were investigated in this research work by selecting soybean oil, glycerol, phthalic anhydride, maleic anhydride, rosin and benzoic acid.

#### b) Synthesis of Novel Resin<sup>7</sup>

Compositions selected for the preparation of alkyd resins are given in table No. 2. The percentage of soybean oil was taken 7. The chain stoppered compound like Benzoic acid and rosin was used in this formulation. The content of maleic anhydride varied between 1 to 4 % while phthalic anhydride varied between 30 to 45 %.

#### A) The Reactor :

The preparation of alkyd resin was carried out in a glass reactor<sup>5</sup>. The reactor consists of two parts. Lower part of the reactor is a round bottom vessel with very wide mouth. The capacity of the flask is about 2 litres. The upper part of the reactor is its lid, having four necks with standard joints.

A motor driven stirrer was inserted in the reactor through the central neck, while another neck was used for thermometer. A condenser was fitted with the reactor through the third neck. And the fourth neck was used for dropping the chemicals in to the reactor. The reactor was heated by a electric heating mantle having special arrangement for smooth control of the temperature of the reactor. The speed of the stirrer was controlled by a regulator. The reaction vessel and its lid were tied together with the help of clamps.

#### B) Stoichiometrical Calculation for individual ingredients in alkyds :

The functional groups (-OH and -COOH) present in the reactants of the batch of 100 gms, can be calculated as follows:

##### (I) Calculation for -OH groups

###### (a) Glycerol

Mol. Wt. of Glycerol = 92.12

One mole Glycerol contains 3 - OH groups

$$\begin{aligned} & 3 \times 286.8 \\ \therefore \text{No. of } -\text{OH groups in 286.8 gms. of glycerol} &= \text{-----} \\ & \text{--} \\ & 92.12 \\ & = \mathbf{9.3399} \end{aligned}$$

**(II) Calculation for –COOH group**

**(a) Rosin**

$$\begin{aligned} & 56100 \\ \text{Mol. Wt. of Rosin} &= \text{-----} \\ & \text{Acid value} \\ \text{Acid value of Rosin is } &166.5 \\ & 56100 \\ \text{Mol. Wt. of Rosin} &= \text{-----} = 336.9 \\ & 166.5 \\ \text{One mole Rosin contains one } -\text{COOH group} & \\ & 1 \times 150 \\ \therefore \text{No. of } -\text{COOH group in 150 gms. of Rosin} &= \text{-----} \\ & 336.9 \\ & = 0.445 \end{aligned}$$

**(b) In Maleic anhydride**

$$\begin{aligned} \text{Mol. Wt. of Maleic anhydride} &= 98 \\ \text{One mole of Maleic anhydride contains 2 } -\text{COOH} & \\ \text{groups.} & \\ \therefore \text{No. of } -\text{COOH group in 30.4 gms. of Maleic anhydride} & \\ = & \\ 2 \times 30.4 & \\ \text{-----} &= \mathbf{0.6204} \\ 98 & \end{aligned}$$

**(c) In phthalic anhydride**

$$\begin{aligned} \text{Mol. Wt. of Phthalic anhydride} &= 148 \\ \text{One mole of Phthalic anhydride contains 2 } -\text{COOH groups.} & \\ \therefore \text{No. of } -\text{COOH group in 416.3 gms. of phthalic anhydride} & \\ = & \\ 2 \times 416.3 & \\ \text{-----} &= \mathbf{5.6256} \\ 148 & \end{aligned}$$

**In Benzoic acid**

$$\begin{aligned} \text{Mol. Wt. of Benzoic acid} &= 122.12 \\ \text{One mole of Benzoic acid contains 1 } -\text{COOH groups.} & \\ \therefore \text{No. of } -\text{COOH group in 50 gms. of Benzoic acid} &= \\ 1 \times 50 & \\ \text{-----} &= \mathbf{0.4094} \\ 122.12 & \end{aligned}$$

$$\begin{aligned} \text{Total No. of } -\text{COOH group} &= \mathbf{7.1004} \\ \text{Total No. of } -\text{OH group} &= \mathbf{9.3399} \\ \text{Excess of } -\text{OH group} &= \mathbf{2.2395} \end{aligned}$$

$$\% \text{ Excess of } -\text{OH group} = \mathbf{31.5}$$

**C) Reaction programming:**

Alkyd resins were prepared by batch process<sup>4</sup>. Heating schedule for alkyd resin is given in flow chart No. 1. The reaction, temperature and addition of ingredients are detailed below: -

**First Step:** The Soybean oil along with rosin, glycerol, maleic anhydride, benzoic acid and solvent (XBT) were added to the reactor. Then all the reactants were mixed thoroughly and heated up to 130 °c.

**Second Step:** Phthalic anhydride was added to the reaction mixture slowly and heated at 220 °c for about 1 hour.

**Third Step:** The reaction mixture now cooled up to 210 °c and temperature is maintained for about 1 hour. Again the mixture was cooled to 200 °c and maintained this temperature for 1 hour.

When the cooking was completed as indicated by acid value, the reaction mixture was cooled up to 100 °c and poured into a pan. Allow the resin to solidify to get the crystals of resin. Pack the crystals in a tin container. Analysis of alkyd resin were carried out for different physicochemical properties, analysis is given in table No. 3.

**Preparation of enamel paint:**

Enamel paint was prepared using short oil alkyd resin and evaluated for their physico-chemical and film properties<sup>8</sup>.

**(i) Composition:** The composition of the enamel paint, to be prepared, was selected in such a way that the paint would contain 27.80% of TiO<sub>2</sub> pigment, 2.78% of ZnO pigment, 0.36 % phthalocyanine blue pigment and 20.20 % of alkyd resin. The paint would have the viscosity of about 100 seconds. The composition of enamel paint, are given in **table no. 4.**

**(ii) Procedure:** The preparations of the enamel paint of the above said compositions were carried in three steps.

**(a) First step:** Preparation of alkyd resin solution of viscosity 60 seconds.

A known amount of solid alkyd resin was taken in a beaker and was diluted with required quantity of solvent (a mixture of xylene and butanol in a ratio of 9:1) to get the the viscosity of 60 seconds. Total weight of alkyd resin solution, obtained, was noted.

**(b) Second Step:** Preparation of paste

56 gms. of Titanium dioxide pigment , 6 gms. of Zinc oxide pigment, 0.72gms. of phthalocyanine blue were taken in a beaker. About 80 gms of alkyd resin solution of viscosity 60 seconds (prepared in first step) was mixed with the pigments. They were then passed through triple roll mill to get desired fineness of grind.

A thick light blue paste was obtained.

**(c) Third step:** Dilution of paste.

The viscous paste, thus obtained, was tested for its viscosity. It was diluted with solvent to reduce the viscosity to 60 seconds. The total quantity of paint, thus prepared, was weighed. The enamel paint thus prepared will have viscosity of 60 seconds and the weight percentage of TiO<sub>2</sub>, ZnO and phtylocycnine blue will be 27 %, 2.7% and 0.36 % respectively.

The enamel paint prepared was analyzed for physicochemical and film properties. These properties are given in table no. 5 and 6. Its resistance to water, acid, alkali, detergent and solvent have been given in Table No.7.

### RESULT AND DISCUSSION

In general, the reported compositions of short oil alkyds have an oil percentage of 25 to 35. If we try to reduce the content oil below this level the control of reaction is lost and there is heavy tendency of gelation of alkyd formulation. If we make use of chain stoppers like Benzoic acid or Rosin, perhaps the proper control of the reaction can be achieved.

Table 2 gives the composition of short oil alkyd using soybean oil. Here soybean oil is specifically chosen because it does not contain higher proportion of linolenic acid. So tendency of gelation can be reduced to some extent by using soybean oil. Rosin is having bulky structure with molecular weight of 300. This is bulkier molecule therefore it will arrest and regulate the growth of polymer. About 5% of Benzoic acid is known to control and to regulate the fast polymerization reaction. There are several distinct advantages in this reaction. First of all, the total time of heating is very less. It is just about 4 to 5 hours. Normally a standard alkyd cook required 12 to 16 hours for cooking therefore there is definite saving of time and energy. Highest temperature required in this process is 220°C. normally while making use of D.C.O. in alkyd we have to cross the temperature of 270 °c. The energy required, fuel cost, color and quality of resin demand that the highest temperature should not go above 220°C.

The analysis of this short oil alkyd is given in table 3. The viscosity of composition is quite high. Drying time is quite satisfactory. The surface dry time is only 15 minutes, which is remarkable property of this composition. Synthetic enamel paint was prepared based on this alkyd using TiO<sub>2</sub>, ZnO as a pigment. The physico-chemical properties are studied in table 5. The sample has high hiding power. The viscosity is quite satisfactory for application by spray or brush. The hiding power of paint is excellent i.e.14 to 15 square meter per liter. The adhesion of paint sample is also excellent. The surface dry time is very less it is just 15 minutes while hard dry time is 6 hours. Scratch hardness of the paint sample is above 1000 grams. The resistance to water, 3% sulphuric acid for 48 hours is excellent while resistance to all detergent and 3 % sodium hydroxide is

poor. Finally, the resistance of paint sample for all the solvents is excellent.

In general, the resistance of paint samples to solvents, 3% detergent, 3% alkali and water is excellent. Resistance in water is good even after 7 days. The only defective part is poor resistance for 3% H<sub>2</sub>SO<sub>4</sub>.

### CONCLUSION

The following conclusion stand confirmed in the light form of the above experimental work.

- 1) Normally, short oil alkyd contains 25 to 35% oil in the final composition. Here we have made an attempt to prepare alkyd with very low oil length. The oil has been kept very low (7 to 15 %). Normally sample gel at such a lower percentage of oil. However by making use of chain stoppers we control reaction in workable range.
- 2) Thus the chain stoppers like benzoic acid and rosin is used to control reaction. Phthalic anhydride is used as major acid ingredient. The polyol used is glycerol.
- 3) The major advantage is saving of time in cooking of batch. Conventionally the alkyd batch required cooking schedule of 12 to 16 hours. While here the complete schedule is of 8 hours. Thus using this composition we can increase the capacity of the plant. Thus overall efficiency of process can be improved.
- 4) In D.C.O. resonated alkyd for dehydration stage we have to increase temperature 275 oc. Thus higher temperature in the range of 270 to 280°C is required. Here in present work, the highest temperature required is 220 °c. Usually we work in lower temperature of 180 to 190 °c. From the viewpoint of controlling reaction, the lower cooking temperature is attractive feature. The energy consumption is also reduced. Looking to the skyrocketing prices of fuel and electricity, a lower temperature is certainly advisable and profitable.
- 5) Useful emulsion paste base on alkyd has been prepared. In the composition emulsion paste distilled water to the extent of 33 to 56 % can be incorporated. The solvent used in paste is smaller quantity of iso propanol, pine oil and moderate quantity of xylene and butanol. A small amount ammonia have been incorporated for the residual acidity of alkyd. A small quantity of P.V.A. 1 to 3 % has been incorporated to improve the homogeneity, adhesion and other film properties of paints. A small quantity of commercial homogeniser 1 to 2 % has been used in emulsion paste. This paste has low acid value and contains 40 to 50 % non-volatile. The paste has stability of more than three month. A small quantity of sodium penta chlo phenol has been incorporated to improve stability. In general, excellent paint composition can be prepared by using 20 to 60 % paste.

- 6) The paint compositions have excellent physico-chemical properties. The fineness of grind, specific gravity, hiding and viscosity is comparable to commercial sample.
- 7) Resistance of film to water even for 7 days is excellent. Many formulations have excellent resistance to detergent and solvent. Acid and alkali resistance needs improvement.
- 8) Most important and attractive feature of this work is thinning paint with organic solvent as well as water. Thus addition of 10 % of organic and 10% of water will not adversely affect consistency of paint.
- 9) Pilot Plant trials and commercial marketing of these products should be undertaken to prove these experimental results.

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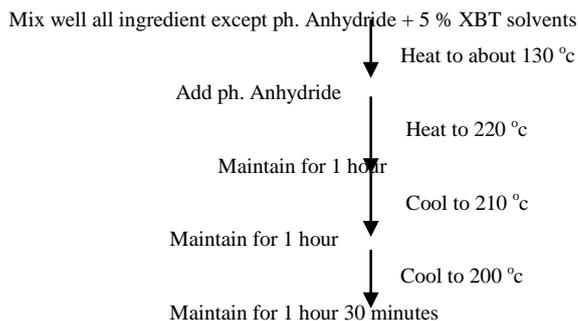
TABLE 1: SOURCE AND ANALYSIS OF RAW MATERIAL/ CHEMICALS.

Raw materials/ chemicals/ reagents	Grade of the Materials	Source of The Materials	Results of Analysis
Soybean oil	Commercial grade	Swastik Acids and Chemicals Nagpur	Acid value = 3 Sap. Value =190 Iodine value = 130 Sp. gravity = 0.923
Rosin	-do-	-do-	Acid value = 166.5
Glycerol	Lab. Chemicals	E. Merch (India) Ltd. Mumbai.	% purity = 98 density =1.255 moisture content = 1.2 %sulfated ash = 0.005
Maleic anhydride	Lab. Chemicals	Swastik Acids and Chemicals Nagpur	Acid value = 1130.0
Phthalic anhydride	Lab. Chemicals	Swastik Acids and Chemicals Nagpur	Acid value = 750.0
Benzoic acid	Lab. Chemicals	Samar Chemicals, Nagpur	% purity = 99 m.p. = 121 °c
Titanium dioxide	Anatase grade	Travancore titanium products Ltd., India	Oil absorption= 21.8%
Zinc oxide	Lab. Chem.	E. Merch. ( India) Ltd. Mumbai	Oil absorption =15.9%
Xylene	Lab. Chem	Loba Chemie Pvt. Ltd. Mumbai	B.P. = 138°c Sp.gr = 0.859
Turpentine	Commercial grade	Swastik Acids and chem. Nagpur	B.P. =158.5°c Sp.gr. = 0.872
Butanol	Lab. Chem	S. Define chem. Ltd. Boisar.	B.P. =116.5oc Sp.gr. = 0.810

TABLE 2: COMPOSITION OF SHORT OIL ALKYD RESIN BASED ON SOYBEAN OIL AND PHTHALIC ANHTDRIDE .

Ingredient	Composition % by weight
Soybean oil	7.00
Glycerol	28.68
Phthalic anhydride	41.63
Maleic anhydride	3.04
Benzoic acid	5.00
Rosin	15.00

**Folw chart No.1. HEATING SCHEDULE FOR SHORT OIL ALKYD BATCH B1.**



**TABLE 3: ANALYSIS OF FINAL SHORT OIL ALKYD SAMPLE.**

Acid value	39
% non volatile	92
Viscosity of 45 % solid using Ford cup no.4 at 30 °c in seconds.	60
Drying time	
Surface dry ( in minutes )	15
Hard dry ( in hours )	5

**TABLE 4. COMPOSITION OF ENAMEL PAINT P1 BASED ON ALKYD RESIN.**

Ingredient	Composition % by weight
Titanium dioxide	27.80
Zinc oxide	2.78
Ph. Green	0.36
Short oil alkyd	20.20
Solvent (xylene: butanol)	48.86

**TABLE 5: PHYSICO-CHEMICAL PROPERTIES OF PAINT BASED ON BATCH B1.**

Fineness of grind in micron	3
Specific gravity	1.29
Viscosity by Ford cup no.4 at 30 °c in seconds	60

**TABLE 6: FILM PROPERTIES OF PAINT P1 BASED ON BATCH B1.**

Drying time	
Surface dry ( in minutes )	15
Hard dry ( in hours )	4
Scratch hardness in grams	1000
Adhesion	Excellent
Gloss	Matt

**TABLE 7: ANALYSIS OF PAINT RESISTANCE TO WATER, ACID, ALKALI, DETERGENT AND SOLVANTS.**

Water	
48 hours	Excellent
7 days	Excellent
Acid ( 3% H2SO4) for 48 hours	Excellent
Alkali ( 3% NaoH) for 15 minutes	Very poor
Detergent 3% for 48 hours	Poor
Solvents ( 15 minutes)	
Xylene	Excellent
Butanol	Excellent
Turpentine	Excellent

Excellent = Film unaffected  
Poor = Film affected

Good = Film slightly affected  
Very poor = Film completely removed