



## STUDY OF DETERGENT POWDER PREPARED FROM NOVEL SHORT OIL ALKYD SYNTHESIZED FROM LINSEED OIL AND ROSIN.

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### ABSTRACT

A novel short oil alkyd has been prepared using materials which are of vegetable origin instead of linear alkyl benzene sulphonate. The price of linear alkyl benzene sulphonate is spirally high every year. Novel short oil alkyd has been prepared by using linseed oil, phthalic anhydride, maleic anhydride and Rosin. The idea of present work is to develop a new polymer based mainly on vegetable sources and use it as an active ingredient in detergent composition. The study involves the combination of SLS, SLES and Dolomite. It has been observed in our experiments that use of high grade dolomite can increase whiteness and brightness to the cloth. Efforts were made to select the optimize proportion of dolomite to get excellent detergency. Various analytical parameters like surface tension, foaming properties and detergency on solid cloths were studied and were found to be at par or sometimes better than commercial samples.

**Key words:** - Alkyd resin, Powder detergent, Sodium lauryl sulphate and Sodium lauryl ethyl sulphate.

### INTRODUCTION:

India<sup>1</sup> is one of the largest producers of soap and detergents in the world. These detergents are well knitted in our daily life. Detergents are made largely from LABS ( Linear alkyl benzene sulphonate). Despite being important to us surfactants have somewhat a tarnished image because of their past association with environmental pollution. So new class of surfactants were introduced that is polymeric surfactant. Most of the polymeric surfactants are biodegradable in nature and prepared from renewable sources. Rosin<sup>2</sup> a major ingredient of alkyd polymeric surfactant is a surface active agent. Authors have successfully used sorbitol<sup>3-6</sup> and starch in liquid and powder detergents. The increased public awareness of environment has created serious concern about the disposal of synthetic polymer. So there is a need to produce safe and novel products to health and environment, which are effective and cheap. The use of linseed based polymer is an

attempt towards it. The experimental conditions were worked out for getting a novel resin based on vegetable products like linseed oil<sup>7</sup> and rosin<sup>8</sup>.

### MATERIAL & METHODS

**1. Synthesis of Alkyd Resin<sup>9</sup>:** Linseed oil, rosin, all the ingredients except phthalic anhydride are taken initially in a standard glass reactor of two litres capacity fitted with tafflon stirrer, thermometer and temperature control arrangement  $\pm 2^{\circ}\text{C}$ . Xylene and 1-butanol (3:1) were used as solvents. Details of heating schedule and order of addition of reactants are given in

Table 1.

### 2. Preparation of Powder Detergent Samples

The various detergent ingredients and the composition of various powder detergents are shown in table 2. The ingredient in the powdered form are weighed and mixed thoroughly in a tray, whole mass is then mixed thoroughly. The mixture is then poured in a mixing pot and worked for twenty minutes. After mixing, the

homogeneous mass thus obtained is taken out in a tray and kept in open air for drying. After complete drying the solid mass thus formed is grinded again in a mixture to get homogenized detergent powder. Ten different types of powder detergent compositions have been prepared as given in table 2. The percentage of resin was varied from

6.5 to 16.5. Sodium lauryl sulphate and Sodium lauryl ether sulphate has been used to have better foaming and cleaning properties. Table No.3 and 4 gives information about surface tension and foaming characteristics. Table 5 and 6 gives information about stain removing of soil and coffee stain at 60°C and 30°C. Our samples are on par or sometimes even better than commercial product which has been tested simultaneously. The testing has been done as per standard methods. Percentage detergency was also determined by using standard soiling technique for cotton and tericot cloth. For measurement of reflectance, standard digital reflectance meter has been used.

### 2.Surface Tension<sup>10</sup>

The surface tension of powder detergent was measured using stalagmometer.

### 3.Foam Volume<sup>11</sup>

Foam is a cause of dispersion of gas in relatively small amount of liquid. This was measured by using mechanical agitation in a closed vessel. Foam characteristics were measured in terms of volume by Bubble cylinder method.

**4.Detergency Test:** This includes the following steps.

#### A. Preparation of soil medium<sup>12</sup>

The soil medium is prepared with following composition. The mixture carbon black

(28.4%)and lauric acid (17.9%)and mineral oil (17.9%) was taken in a pestle mortal and grind thoroughly for 1-2 hours to get fine grinding and smooth filling. About 2gm of above paste was mixed well with 500ml of carbon tetra chloride and used for soiling of fabrics

#### B. Fabric Washing<sup>13</sup>

The solution of 1% concentration of powder detergents in distilled water was prepared. These solutions were heated to 60% and stained fabrics were dipped in it for five minutes. Then fabrics were hand washed with equal strokes. After washing the test materials were rinsed in running tap water, dried and ironed. The same experiment was carried in exactly same manner with commercial detergent sample. The % detergency was found out by using Lambert and Sanders formula.

$$\% \text{ Detergency} = \frac{(R_w - R_s)}{(R_o - R_s)} \times 100$$

Where,  $R_w$  = Washed fabric

$R_s$  = Soiled fabric and

$R_o$ =Standard original fabric.

The reflectance was measured with an electro reflectance photometer with filter R-46 and calibrated against MgO standard.

### RESULT & DISCUSSION:

1. The composition of novel polymer is given in Table No.1. All the raw materials used for the preparation of alkyd resin have percentage purity of 98-99%.This resin was analyzed systematically for their physicochemical characteristics. The special feature is that we have taken oil length that is at 17% and reaction was controlled by using rosin and benzoic acid as chain stoppers. The cooking schedule has been standardized. The time of heating was 9:30 hours.
2. The composition of Powder detergents is given in Table No 2.The concentration of polymers

is varied. All samples contain sodium lauryl sulphate and sodium lauryl ether sulphate. The use of these ingredient helps in getting good foaming characteristics and excellent cleaning on terricot and cotton cloth. Sodium sulphate and urea have been used in small quantity .A small amount of EDTA has been used to make the detergent so that it can work in hard water. Some samples have higher moisture content.

3. The foam volume measured at various concentrations is given in Table No 4. The foaming capacity was found to be appreciable. The detergents give excellent detergency from 60-90%. Various cloths like cotton and tericot give positive results for coffee and soil stain removal.

#### CONCLUSIONS:

The following conclusions stand confirm in the light of above experimental work.

A novel polymer can be prepared based on rosin, linseed oil, maleic anhydride and glycerol. The order of reaction, time of heating, cooking schedule and catalyst has been standardized. The method of cooking is simple, easy to operate and without complication. Normally polymer cooking requires heating schedule of 12-15 hours while our heating schedule is of 9 hours 30 minutes. This saves time and energy as well as the manufacturing cost is less. Synergistic composition of novel resin with conventional active materials like SLS and SLES have been used for special cleaning purpose. Some composition with higher proportion of novel resin is used. No SLS and SLES is used in PD6 – PD10. Thus in these only novel resin is used. These samples are giving very low foam. They have excellent stain removing capacities

for soil and coffee. These detergents will have special feature of water saving.

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**Table.1: Composition and Heating Schedule of Novel Polymer**

Ingredients	% by weight	Order of addition of reactants	Time of heating in Hrs: Mins
Linseed oil	17.0	Linseed oil, rosin, glycerol, catalyst, maleic anhydride, benzoic acid.	
Rosin	34.0	Heat at 170°C	1:00
Glycerol	25.0	Heating at 248°C	2:10
Maleic anhydride	5.0	Cool to 230°C	0:20
Phthalic anhydride	14.0	Slowly cool to 80°C	1:00
Benzoic acid	3.0	Stage B:-Add phthalic anhydride and 5% solvents (3:1) Xylene: Butanol. Heat to 225	1:00
Sodium bisulphate	1.5	Continue reaction further at 225°C	4:00
Sodium bisulphite	0.5	Cool to 80°C and remove the product	
% Yield	93	Total time in Hrs:mins	9:30

**Table.2: Formulation of Detergent Powders Based on Novel Polymer**

Ingredients	PD1	PD2	PD3	PD4	PD5	PD6	PD7	PD8	PD9	PD10
Sodium carbonate	45.0	41.0	38.0	33.2	27.9	40.9	38.1	35.3	31.9	27.0
Resin	10.9	10.0	7.4	7.7	6.5	6.5	6.0	13.4	12.6	10.7
STPP	4.2	3.8	3.4	2.9	2.5	4.3	3.1	3.5	3.1	2.7
EDTA	0.07	0.06	0.06	0.05	0.04	0.08	0.04	.05	0.05	0.04
Sorbitol	2.6	2.3	2.1	1.7	1.5	2.5	2.1	1.9	1.5	1.4
Sodium sulphate	2.6	2.3	2.1	1.7	1.5	2.5	2.1	1.9	1.8	1.4
Urea	1.6	1.5	1.3	1.2	0.9	1.6	1.4	1.2	1.1	0.9
SLS	4.2	3.8	3.4	3.0	2.0	-	-	-	-	-
SLES	2.6	2.3	2.1	1.7	1.5	-	-	-	-	-
Dolomite	8.1	17.1	26.3	35.2	45.2	8.0	16.6	25.5	33.6	43.3
Moisture	18.2	15.9	13.9	11.7	10.5	23.7	20.2	17.3	14.4	12.6

**ABBREVIATIONS**

SLS-Sodium lauryl sulphate SLES- Sodium lauryl ether sulphate

**Table.3: Analysis of Surface tension of powder Detergents (dynes/cm)**

Samples	0.1	0.25	0.5	1.0
PD1	44.6	39.3	34.4	31.2
PD2	50.1	43.5	39.4	34.1
PD3	48.6	45.3	38.1	31.5
PD4	69.5	61.8	51.1	44.7
PD5	50.5	41.2	37.2	32.8
PD6	68.7	53.4	58.7	47.8
PD7	43.5	36.3	32.2	30.5
PD8	66.2	63.5	55.5	53.7
PD9	67.7	65.0	62.4	5.3
PD10	62.8	65.0	60.4	52.6
Commercial	29.5	26.5	24.1	22.8

**Table.4: Foam Volume of Powder Detergents**

Sample s	Foam Volume (0.1%) Time in minutes				Foam Volume (0.25%) Time in minutes				Foam Volume (0.5%) Time in minutes				Foam Volume (1%) Time in minutes			
	0	5	10	15	0	5	10	15	0	5	10	15	0	5	10	15
PD1	50	45	40	30	60	55	55	40	120	110	100	90	150	140	130	120
PD2	60	50	40	30	100	90	80	70	120	110	100	90	150	140	130	125
PD3	70	65	60	50	100	90	80	70	130	120	110	100	160	150	140	130
PD4	50	40	30	20	100	110	100	90	120	120	110	100	140	130	120	110
PD5	40	30	20	10	60	50	40	30	100	90	80	70	150	140	130	120
PD6	10	10	05	05	10	10	05	05	20	10	05	05	30	20	15	10
PD7	20	10	05	05	30	5	20	15	40	30	20	15	40	30	20	50
PD8	40	30	25	20	50	40	30	20	50	40	30	20	60	50	40	30
PD9	20	15	10	10	40	30	20	15	40	30	20	10	40	50	30	20
PD10	20	20	10	10	30	20	10	10	40	25	20	20	40	35	30	20
commer cial	170	150	130	120	160	150	140	130	140	130	130	120	310	210	180	160

**Table.5: Evaluation of Detergency**

Samples	For Soil Stain				For coffee stain			
	Cotton (% detergency)		Tericot (% detergency)		Cotton (% detergency)		Tericot (% detergency)	
	0.5	1	0.5	1	0.5	1	0.5	1
PD1	74.1	75.2	91.0	91.8	63.1	64.0	84.8	81.2
PD2	75.5	77.8	80.0	87.6	72.2	73.1	86.0	87.6
PD3	85.8	89.4	78.7	88.1	70.4	76.3	83.2	90.4
PD4	67.8	73.0	93.1	69.2	76.8	80.9	86.8	82.8
PD5	71.9	78.4	69.2	77.6	70.0	75.9	86.0	90.0
PD6	70.5	73.0	85.2	87.6	70.4	70.9	83.6	84.0
PD7	76.5	79.0	86.3	86.5	63.1	70.4	93.6	94.0
PD8	78.7	81.1	82.1	85.2	71.8	72.2	93.2	94.8
PD9	72.7	75.7	74.7	80.5	72.7	73.6	87.6	88.8
PD10	68.6	70.0	70.5	71.3	72.2	72.2	78.4	80.4
Commercial	76.5	83.3	80.5	85.7	72.2	84.0	91.6	87.6