



LAUNDRY DETERGENTS BASED ON NOVEL POLYMERIC SURFACTANTS CONTAINING LINSEED OIL AND ROSIN

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Abstract: In conventional alkyd polymer 40-50% Phthalic anhydride is utilized in short oil compositions. We are interested in developing polymer based mainly on vegetable resources rather than the petroleum products like phthalic anhydride. In view of the above, a novel alkyd polymer containing vegetable plant based linseed oil and rosin has been prepared and used as partial replacement of linear alkyl benzene sulphonate in detergent. 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 soiled cloths were studied and were found to be at par or sometimes better than commercial samples. Thus above prepared novel alkyd polymer based surfactant that is free from phosphates and other harmful ingredients may be technically superior, economically attractive and ecofriendly substitutes for conventional detergents based on petroleum products.

Keywords: Alkyd resin, Powder detergent, linseed oil, rosin, Sodium lauryl sulphate and Sodium lauryl ethyl sulphate

I. INTRODUCTION

The extensive usage of petroleum products develops a global pressure on petroleum products. Moreover, these materials are big threat to the safe and healthy environment. In these circumstances we restrict the use of petroleum products by replacing them with renewable resources of natural origin which are biodegradable and ecofriendly. In India a very vast renewable vegetable sources such as plants, oilseeds, starch and fruits are available. Many of these, when modified can be used as suitable substitutes for petroleum products.

India¹ 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.

Linseed oil² also known as flaxseed oil is a clear to yellowish oil obtained from dried ripe seeds of the flax plant.

The linseed oil³ is enriched in alpha linolenic acid, an n-3 polyunsaturated fatty acid (PUFA), which is important nutritionally as well as prone to oxidation and hence is a drying oil with many industrial uses.

Rosin⁴, or colophony, is a natural, abundant, cheap and non-toxic raw material which can be easily modified to obtain numerous useful products, which makes it an excellent subject of innovative research, attracting growing interest in recent years.

Rosin⁵ a major ingredient of alkyd polymeric surfactant is a surface active agent. Authors have successfully used sorbitol⁶⁻⁹ 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 oil¹⁰ and rosin¹¹

II. Experimental:

1. Synthesis of Alkyd Resin¹²: 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 mixer 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 6.0 to 13.4. A very less amount of Sodium lauryl sulphate and Sodium lauryl ether sulphate has been used in only 05 samples (PD1 to PD5) and another 05 samples prepared without using SLS ND SLES. (PD6 to PD10).

2. Surface Tension¹³

The surface tension of powder detergent was measured using stalagmometer.

3. Foam Volume¹⁴

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 medium¹⁵

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 mortar and grind thoroughly for 1-2 hours to get fine grinding and smooth filling. About 2gm of above paste was mixed well with 500 ml of carbon tetra chloride and used for soiling of fabrics.

B. Fabric Washing¹⁶

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.

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

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.

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 ^o C	1:00
Glycerol	25.0	Heating at 240 ^o C	2:10
Maleic anhydride	5.0	Cool to 230 ^o C	0:20
Phthalic anhydride	14.0	Slowly cool to 80 ^o 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 ^o C	4:00
Sodium bisulphite	0.5	Cool to 80 ^o 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	46.0	42.0	39.0	34.2	28.9	41.9	39.1	36.3	32.9	28.0
Resin	10.9	10.0	7.4	7.7	6.5	6.5	6.0	13.4	12.6	10.7
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	17.2	14.9	12.9	10.7	9.5	22.7	19.2	16.3	13.4	11.6

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	46.6	37.3	35.4	32.2
PD2	52.1	44.5	41.4	36.1
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	51.3
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

Samples	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	12	11	10	90	17	15	16	120
PD2	60	50	40	30	10	90	80	70	12	11	10	90	15	14	13	125
PD3	70	65	60	50	10	90	80	70	13	12	11	100	16	15	14	130
PD4	50	40	30	20	10	11	10	90	12	12	11	100	15	14	13	110
PD5	40	30	20	10	60	50	40	30	10	90	80	70	16	15	14	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
Commercial Sample	170	150	13	12	16	15	14	13	14	13	13	120	31	21	18	160

Table.5: Evaluation of Detergency

Samples	For Soil Stain				For coffee stain			
	Cotton (% detergency)		Terricot (% detergency)		Cotton (% detergency)		Terricot (% detergency)	
	0.5	1	0.5	1	0.5	1	0.5	1
PD1	73.1	76.2	92.0	93.8	65.1	66.0	87.8	89.2
PD2	74.5	77.8	80.0	87.6	72.2	73.1	86.0	87.6
PD3	84.8	89.4	78.7	88.1	70.4	76.3	83.2	90.4
PD4	66.8	73.0	93.1	69.2	76.8	80.9	86.8	82.8
PD5	72.9	78.4	69.2	77.6	70.0	75.9	86.0	90.0
PD6	71.5	73.0	85.2	87.6	70.4	70.9	83.6	84.0
PD7	77.5	79.0	86.3	86.5	63.1	70.4	93.6	94.0
PD8	79.7	81.1	82.1	85.2	71.8	72.2	93.2	94.8
PD9	74.7	75.7	74.7	80.5	72.7	73.6	87.6	88.8
PD10	69.6	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	81.6	87.6

III. Results and 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. Out of the Ten sample, first 05 samples contain sodium lauryl sulphate and sodium lauryl ether sulphate. 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. 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 terricot cloth. For measurement of reflectance, standard digital reflectance meter has been used.
4. 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 terricot give positive results for coffee and soil stain removal.

IV. Conclusion:

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. Comparative study of samples were done, in that increasing the amount of alkyd resin by decreasing amount of SLS without sacrificing technical properties of detergents. Some composition with higher proportion of novel resin is used. SLS and SLES were not added in PD6 – PD10, 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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