



MODERATE FOAMING ECOFRIENDLY DETERGENTS BASED ON ROSIN AND LINSEED OIL

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Abstract

Novel polymers based on linseed oil, phthalic and maleic anhydride have been synthesized. The variation in mole ratios, reaction temperature, type of catalyst and the time of heating has been studied. The idea of the present work is to develop a new polymer based mainly on vegetable sources and use it as an active ingredient in detergent composition. An ecofriendly alkyd resin polymer based on linseed oil and rosin was synthesized and used with Sodium Lauryl Sulphate (SLS) instead of LABS for detergent formulation. Efforts are made to select the optimize proportion of dolomite to get excellent detergency. Based on surface tension, detergency and foam volume the prepared compositions are on par or sometimes better than commercial samples. The proposed composition can be easily prepared in existing alkyd resin manufacturing plant with little modifications.

Keywords

Alkyd resin, polymeric surfactant, powder detergent.

Introduction

The price and availability of alkyl benzene sulphonate which is basically of petroleum origin is soaring every year because of crude petroleum prices. We have to search for alternative active materials which are of vegetable origin.

India has a vast capacity of manufacturing alkyd resin which is used in surface coating industries. The per capita consumption of detergent is higher than surface coating (paint industry), therefore the same alkyd resin plants can produce alkyds suitable for surfactants in the same set up without much investment. A novel polymeric surfactant based on linseed oil¹, maleic anhydride², and rosin³ has been synthesized. Technically it is a rosinated short oil alkyd resin⁴. Short oil alkyd resin⁵ has been successfully used as polymeric surfactants in various powder and liquid detergents. The reduction and removal of polyphosphates for getting ecofriendly detergents is only





possible because of polymeric surfactants. Earlier polymeric surfactants were used only as an additive while we used resin as a total substitute of linear alkyl benzene sulphonate (LABS) successfully. Rosin a major ingredient of alkyd resin polymeric surfactant is a surface –active agent⁶. The present work is aimed at total replacement of LABS with polymeric surfactant. It utilizes the surfactant based on polymer for detergency and suggests this as an alternative product as a diversification for existing alkyd resin plants without much additional investment. The special feature of our polymer is the use of 30 to 40% rosin which is abundantly available. The other ingredients are minor proportions of linseed oil 17%, Glycerol 25%, maleic anhydride 5% and phthalic anhydride 14%. The combined use of rosin and linseed oil gives desirable foaming and cleaning properties. The overall idea is to develop ecofriendly detergents for green environment which are free from petroleum products and sodium tripolyphosphate.

Material and Method

Experimental Synthesis of alkyd resin⁷:- 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 $\pm 2000\text{C}$. Xylene and 1-butanol (3:1) were used as solvents. Details of heating schedule and order of addition of reactants are given in table 2. Preparation of powder detergent samples:- The various detergent ingredients and the composition of various detergents powders 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. This mixture is then poured in a mixing pot and worked for 20 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 ground again in a mixer to get homogenized detergent powder. Four different powder detergents compositions have been prepared as detailed in table 3. Powder detergents were prepared by





varying the percentage of resin from 5 to 22%. Small amount of Sodium lauryl sulphate⁸ and Sodium lauryl ether sulphate has been used to have better foaming and cleaning properties. Table No. (4) and (5) gives information about foaming characteristics, surface tension and stain removing of soil and tea stains. 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. Surface Tension⁹:- The surface tension of powder detergent was measured using stalagmometer. Foam volume¹⁰:- 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. Detergency Test:- This includes the following steps. Preparation of soil medium¹¹:- 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 2 gm of above paste was mixed well with 500 ml of carbon tetra chloride and used for soiling of fabrics.. Fabric Washing¹²:- 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 to and fro hand washes were given with equal strokes. After washing the test materials were rinsed in running tap water, dried and ironed. The same experiment was carried in exactly the same manner with commercial detergent sample..The % detergency was found out by using Lambert and Sanders formula. % Detergency = $\frac{R_w - R_s}{R_o - R_s} \times 100$ Where R_w , R_s and R_o are the reflectance measured on washed fabric, Soiled fabric and standard original fabrics respectively. The reflectance was measured with an electro reflectance photometer with filter R-46 and calibrated against MgO standard.





ABBREVIATIONS:- SLS - Sodium Lauryl Sulphate SLES - Sodium Lauryl Ether Sulphate EDTA - Ethylene Diamine Tetra Acetic Acid STPP - Sodium Tripolyphosphate

Result and Discussion

RESULTS AND DISCUSSION 1) The composition of novel polymer is given in table No 1. A large quantity of rosin has been used. Rosin gives good foam, solubility and brilliant appearance to various soap and detergent compositions. Large proportion of rosin will also act as a chain stopper for alkyd synthesis. The order of addition, cooking schedule, amount of catalysts and time of heating has been standardized as given in table No-2. The time of heating was 8:30 hours. 2) The composition of detergent powders is given in table No-3. In composition 57% Sodium carbonate is used. In successive samples dolomite replaces sodium carbonate. Thus from formulations D1 to D5 increasing amount of dolomite is used i.e. 9-47%. The proportion of resin has been changed from 3-5%. Sorbitol helps to give smooth and pleasant feel. 3) All samples contain 1-3% sodium lauryl sulphate and sodium lauryl ether sulphate. This helps in getting good foaming and cleaning characteristics. Both ingredients are of vegetable source and they help us to avoid use of linear alkyl benzene sulphonate which is of petroleum origin. 4) Sodium sulphate and urea has been used in small quantity of 1-3%. 5) The foam volume measured at various concentrations is given in table No 4. The samples have moderate foaming capacity but reduction in surface tension is appreciable. The detergents give excellent detergency from 80-90%. The data indicates that rosinated novel polymers has all the desired characteristics at 0.25%. Various cloths like cotton and tericot give positive results.





Conclusion

The following conclusions stand confirm in the light of above experimental work 1) A novel polymer can be synthesized 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 complications. Normally polymer cooking requires heating schedule of 12-15 hours while our heating schedule is of 8 hours. This is certainly saving time and energy. 2) Many alkyd resin plants are lying idle and not in use. These alkyd resin plants can produce alkyds in the same set up without much investment. 3) We are able to search moderate foaming detergents which have excellent reduction in surface tension and cleaning characteristics. These moderate foaming powder detergents can also be used in washing machine, floor cleansers, and also other industrial operations where foam is not needed. These ecofriendly samples have excellent detergency characteristics comparable to commercial samples. Thus they save water. 4) Sample D4 is the best moderate foaming detergent prepared. It contains very less percentage of S.L.S and SLES therefore the cost is less and the cheapest of all the powder detergents. So it is recommended for commercial. 5) All the samples contain less percentage of Sodium tripolyphosphate which promotes foaming eutrofication and spoilage of water and environment.

Acknowledgement

The author is thankful to Dr. B. B. Gogte, Principal, Agnihotri Engineering College, Wardha for his kind co-operation and valuable guidance.

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Table 1: Composition of novel polymer

Ingredients	% by weight
Linseed oil	17.0
Rosin	34.0
Glycerol	25.0
Maleic anhydride	5.0
Phthalic anhydride	14.0
Benzoic acid	3.0
Sodium bisulphate	1.5
Sodium bisulphite	0.5
% Yield	93





Analysis of novel polymer

Property	Observations
Acid value	20.2
% solid	92
Viscosity	120

Table 2: Heating schedule for resin cooking

Order of addition of reactants	Time of heating in Hrs: Mins
Linseed oil, rosin, glycerol, catalyst, maleic anhydride, benzoic acid.	
Heat at 170 ^o C	1:00
Heating at 24 ^o 0C	2:10
Cool to 230 ^o C	0:20
Slowly cool to 80 ^o C	1:00
Stage B:-Add phthalic anhydride and 5% solvents (3:1) Xylene: Butanol. Heat to 225	1:00
Continue reaction further at 225 ^o C	3:00
Cool to 80 ^o C and remove the product	
Total time in Hrs: Mins	8:30

Table 3: Formulation of detergent composition using Dolomite

Ingredients	D1	D2	D3	D4	D5
Sodium carbonate	48.3	45.0	39.3	34.2	29.6
STPP	5.1	4.7	4.1	3.8	3.0
EDTA	0.07	0.07	0.06	0.05	0.04
Resin	9.7	8.9	7.8	6.8	5.7
Sorbitol	2.7	2.5	2.3	2.0	1.6
Sodium sulphate	2.7	2.5	2.3	2.0	1.6
Urea	1.9	1.6	1.4	1.3	1.1
SLS	2.7	2.4	2.3	2.0	1.6
SLES	2.7	2.4	2.3	2.0	1.6
Dolomite	8.6	17.8	26.9	36.3	46.2
Moisture	15.6	12.2	11.3	9.9	8.0





Table 4: Study of surface tension and foam volume of detergent samples.

Foam is measured using standard cylindrical method and expressed in terms of Volume Cm^3 .

Detergent Samples	Surface tension (dyne/cm)	% reduction in surface tension	Foam volume at 0.25%	
			0 mins	15 mins
D1	43.7	39.3	20	20
D2	48.8	32.1	40	25
D3	46.4	35.6	50	30
D4	41.3	42.6	30	15
D5	57.5	20.1	100	70
Commercial	23.8	68.8	160	140

Table 5:-Evaluation of detergency at 1% concentration.

Detergent samples	For Tea Stain		For Soil Stain	
	Cotton (% detergency)	Tericot (% detergency)	Cotton (% detergency)	Tericot (% detergency)
D1	81.7	88.2	85.5	73.6
D2	76.6	81.0	80.3	62.8
D3	78.4	70.2	77.1	72.1
D4	83.7	85.1	87.4	70.7
D5	83.3	88.7	82.5	76.8
Commercial	94.0	95.1	97.4	95.0

