The Asian Journal of Experimental Chemistry, (June, 2011); Vol. 6 Issue (1) : 17-21

Research Paper :

A novel short oil alkyd resin based on coconut oil and rosin for detergent compositions

■PRAVIN A. DHAKITE, **B.B. GOGTE AND** B.W. PHATE

See end of the paper for authors' affiliations

Correspondence to:

B.B. GOGTE
Department of Applied Chemistry, Shri Shankarprasad Agnihotri College of Engineering, Ramnagar, WARDHA (M.S.) INDIA
Email : pravinchemkb@rediffmail.com

ABSTRACT

Alkyd resin is normal raw material for paints, printing inks, and water thinable coatings. In the present work, we have successfully prepared a very short oil alkyd using high proportion of rosin, the other ingredient are coconut oil, maleic anhydride and glycerol. Various parameters like temp., time of heating and mole ratio have been studied. All the sample have been analyzed for physiochemical characteristics like acid value saponification Value, HLB ratio, viscosity, and cleaning performance on soil cloth. Two samples AR-1, AR-2, have given good result so these samples have been used after neutralization with KOH in powder detergent and liquid detergent compositions. These polymer are based on vegetable origin like coconut oil, glycerol and rosin. The above result indicates that novel short oil alkyd can be used as replacement of crude petroleum based acid slurry and Alpha Olefin Sulphonate. The use of coconut oil and rosin gives good foaming and cleaning properties. The price of this material is slightly lower than conventional petroleum based actives. This product will also support green chemistry as the raw materials are of vegetable origin.

KEY WORDS : Alkyd polymer, Detergent powder, Detergency evaluation, Alkyd resin, Resin

How to cite this paper : Dhakite, Pravin A., Gogte, B.B. and Phate, B.W. (2011). A novel short oil alkyd resin based on coconut oil and rosin for detergent compositions. *Asian J. Exp. Chem.*, **6** (1): 17-21.

Received : *04.012011;* **Revised :** *20.03.2011;* **Accepted :** *05.04.2011*

A novel polymeric surfactant based on coconut oil[1], maleic anhydride[2] and rosin[3] has been synthesized, technically it is a rosinated short oil alkyd resin[4]. Short oil alkyd resin has been successfully used as polymeric surfactants in various powder and liquid detergents. In the piece of research work, the mole ratio, catalyst and heating schedule has been standardized to get on alkyd resin with higher acid value, desired hydrophilic-lipophilic ratio (HLB)[5], Viscosity and solubility characteristics.

In the present work, an effort has been made to replace crude petroleum based acid slurry and alpha olefin sulphonate (AOS) with novel polymers. The composition of two selected polymers was found to be useful for powder and liquid detergents (Table 1). The special feature of our polymers is used of 50 to 60 per cent rosin which is abundantly available and prices are quite stable. The other ingredients are minor proportions *i.e.* coconut oil 25 to 30 per cent , glycerol 10 per cent and maleic anhydride 7.5 per cent. The combined used of rosin and coconut oil gives good foaming and cleaning properties.

EXPERIMENTAL METHODOLOGY

Experimental:

Preparation of short of alkyd resin :

This resin was prepared in a three neck glass reactor fitted with stirrer and condenser and temperature control of + 2 ^{0}C . A novel catalyst 1.5 per cent sodium bisulphate and 0.5 per cent sodium bisulphate has been used for this reaction. The composition, cooking schedule and analysis of alkyd polymer is given in Table 2.

Preparation of detergent powders:

Detergent powder was prepared in a laboratory blender mixer and finally moisture content of 14-15 per cent has been maintained in finished powder. Two samples of detergent powder have been prepared one based totally on an alkyd polymer and in other composition 50 per cent of alkyd polymer has been replaced by alpha olefin sulphonate and acid slurry.

Surface tension[6]:

The surface tension of powder detergents was measured using stalagnometer.

Table 1 : Composition of alkyd polymer

Sr. No.	Ingredient	% By weight	
		AR1	AR 2
1.	Coconut oil	30	25
2.	Maleic anhydride	7.5	7.5
3.	Resin	52.5	57.5
4.	Glycerol	10	10

Catalyst used: 1.5% sodium bisulphate and 0.5% sodium bisulphite

Table 2 : Cooking schedule of alkyd polymer

Sr. No.	Time (Hr)	Temp. (^{0}C)
1.	0.00— 2.00	130
2.	2.00— 4.00	225
3.	4.00— 6.00	200
4.	6.00— 8.00	200

Foam volume[7]:

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

Detergency test:

This includes the following steps:

Preparation of soil medium[8]:

The soil medium was prepared with following composition. The mixture of carbon black (28.4%) and laurel acid (17.9%) along with mineral oil (17.9%) was taken in a pestle mortal for 1-2h to get fine grinding and smooth filling. About 2g of above paste was mixed well with 500ml of carbon tetrachloride and for soiling of fabrics.

Fabrics washing[9]:

The solutions of 1 per cent concentration of powder detergents in tap water were prepared. These solutions were heated to 60^{0}C and stained fabrics were dipped in it for five minutes. Ten to and fro hand washes in tap water were given with equal strokes. After washing, the test materials were rinsed in running tap water, dried and ironed. It was also tried with commercial powder detergent. After, the %detergency was found out by using Lambert and Sanders formula:

$$\% \text{ Detergency} = \frac{(Rw - Rs) \times 100}{(Ro - Rs)}$$

where Rw, Rs and Ro are the reflectance measured on washed fabrics, stained fabrics (before washing) and clean fabrics, respectively. The reflectance was measured with an elrepho reflectance photometer with filter R-46 against an MgO-standard.

EXPERIMENTAL FINDINGS AND ANALYSIS

The composition of short oil alkyd is given in Table 1, A large quantity of rosin has been used. Rosin gives good foam, solubility and brilliant appearance to various soap and detergent composition. However we are using large proportion of rosin, which will act as a chain stopper for alkyd and play a positive part in detergent functioning. The cooking schedule has been standardized. The time of heating is 8 hours Analysis of resin is given in Table 3.

Table 3 : Analysis of alkyd polymers

Sr. No.	Analysis	Result	
		AR1	AR 2
1.	Acid value	28.71	32.57
2.	Color	Dark brown	Dark brown
3.	Consistency	thick	Thick
4.	% solid	83	84%
5.	HLB	13.21	13.20
6.	pH	4.7	4.42
7.	Solubility	CCl_4	CCl_4
8.	Mol. Wt.	5,956	5,551
9.	Viscosity	293	281
10.	Saponification value	320.571	352.09

The composition of detergent powder is given in Table 4 the concentration of alkyd resin has been varied from 10 to 20 per cent . In the subsequent compositions alkyds has been replaced by 50 per cent alpha olefin sulphonate maintaining the concentration. The concentration of all other ingredients has been maintained at a constant level EDTA helps in Ca and Mg sequisterizetion , sorbitol gives smooth and pleasant feel to the powder.

The foam volume measured at various concentrations is given in Table 7. The samples totally based an alkyd have a low foaming capacity but reduction in surface tension is appreciable. The alkyd based formulations can also give an excellent detergency from 88 to 98 per cent . This data certainty indicates that rosinated novel alkyd has all the desire capabilities at 0.25 per cent concentration. Various cloths like terricot, cotton and polyester give the same positive results.

Use of 50 per cent alpha olefin sulphonate gives good foam height, further reduction in surface tension and excellent stain removing properties. A perfect synergism is observed at 50:50 levels of alpha olefin sulphonate and

Table 4 : Composition of powder detergents

Sr. No.	Ingredient	Sample (% by weight)							
		PD1	PD2	PD3	PD4	PD5	PD6	PD7	PD8
1.	Sodium carbonate	10	10	10	10	35	50	50	50
2.	Alkyd Polymer (AR-1)	-	6	12	5	10	-	-	-
3.	Alkyd Polymer (AR-2)	-	-	-	-	-	-	6	12
4.	Acid slurry	12	6	--	5	--	-	-	-
5.	Sodium laurel sulphate	5	5	5	5	5	-	-	-
6.	Sodium silicate	--	--	--	3	3	-	-	-
7.	Sodium sulphate	30	30	30	15	15	14	14	14
8.	Dolomite	20	20	20	14	14	-	-	-
9.	Salt	12	12	12	10	10	10	10	10
10.	Sodium tripolyphosphate	3	3	3	--	--	5	5	5
11.	Urea	--	--	--	--	--	5	5	5
12.	Additives	2.5	2.5	2.5	2.5	2.5			
13.	EDTA	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
14.	CMC						0.5	0.5	0.5
15.	Alpha olefin sulphonate	5	5	5	5	5	12	6	-

Abbreviations: EDTA- Ethylene diamine tetra acitic acid CMC-Carboxyl methyl cellulose

novel alkyd.

The detergent powder based on acid slurry and alkyd resin also gives well Foam height and excellent stain removing properties[10]. Detergent powder totally based on alkyd resin give reduction in surface tension and effective at lower dosage of detergent (0.1%).

Table 5 : Individual per cent detergency evaluation of alkyd resins

Cloth	Medium for staining	Conc. in %	% detergency of alkyd resin	
			AR1	AR 2
Cotton	Soil solution	0.1%	54	63.32
		0.25%	44	67.76
		0.5%	66	71.46
		1.0%	84.61	83.33
		2.0%	78.46	81.66
		3.0%	73.84	78.12

Conclusion:

The following conclusions may be drawn in the light of above experimental work

– A useful novel alkyd of desired molecular weight and acid value[11] can be prepared based on higher proportions of resin and smaller proportions of coconut 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.

– The physicochemical analysis of alkyd polymer[12] shows that this is an alkyd polymer with acid value 28 to 35 and HLB ratio of 13 to 14. The neutralization of this

Table 6 : Per cent detergency evaluation

Cloth	Medium for staining	Conc. in %.	% Detergency by detergent powder								
			PD1	PD2	PD3	PD4	PD5	PD6	PD7	PD8	CD1
Cotton	Soil solution	0.1	71.50	78.86	77.52	82.12	81.75	83.31	82.37	79.79	80.12
		0.25	73.84	79.12	78.32	83.10	83.67	85.11	84.31	81.17	83.31
		0.5	78.46	81.86	80.32	85.32	84.01	86.32	86.17	83.27	85.00
		1.0	84.61	83.33	81.66	87	86.12	87.67	88.12	85.11	81.77
	Tea solution	0.1	89.25	89.56	90.63	89.74	89.88	90.56	90.25	89.25	89.25
		0.25	89.56	89.23	89.22	89.54	89.74	89.45	89.75	89.85	90.12
		0.5	89.75	89.56	89.96	89.45	89.74	88.56	89.69	89.54	89.23
		1.0	90.12	91.23	91.45	93.12	92.56	93.45	93.25	94.23	93.84
	Coffee	0.1	84.23	85.23	84.21	86.25	85.56	89.23	86.23	85.40	84.25
	solution	0.25	85.23	84.56	85.25	85.56	85.65	85.54	85.56	84.25	85.23
		0.5	86.77	89.75	88.23	87.54	88.56	88.23	87.23	88.23	89.21
		1.0	89.56	89.23	90.23	91.25	91.58	92.65	93.14	92.45	93.12

Table 7 : Analysis of detergent powders

Powder detergent	Concentration in %	Foam volume				pH	Surface tension (dyne/cm)
		0	5	10	15		
PD -1	0.1	600	600	550	500	8.5	24.26
	0.25	600	600	550	500	8.0	23.85
	0.5	650	650	600	600	8.0	21.21
	1	700	700	650	600	8.0	20.92
PD-2	0.1	500	450	450	400	8.0	30.12
	0.25	500	450	400	350	8.0	29.20
	0.5	550	500	450	400	8.5	28.96
	1	600	550	500	450	8.5	28.26
PD-3	0.1	450	400	400	300	8.0	29.16
	0.25	450	400	400	400	8.0	28.35
	0.5	500	500	450	400	8.5	27.98
	1	600	600	550	500	8.5	27.29
PD-4	0.1	400	400	350	300	8.0	34.34
	0.25	450	400	400	350	8.0	33.78
	0.5	500	500	450	450	8.5	32.64
	1	600	600	550	450	8.5	31.98
PD-5	0.1	400	350	350	300	7.5	35.01
	0.25	450	400	350	300	7.5	34.18
	0.5	500	450	400	350	8.0	33.31
	1	500	550	500	450	8.0	32.78
PD-6	0.1	450	400	400	300	7.5	42.45
	0.25	500	450	450	400	7.5	41.89
	0.5	550	550	450	400	8.0	41.11
	1	600	550	550	500	8.0	40.12
PD-7	0.1	400	350	350	300	7.5	42.88
	0.25	400	350	300	300	7.5	42.07
	0.5	450	400	350	300	8.0	41.51
	1	500	450	400	350	8.0	40.97
PD-8	0.1	350	300	300	250	7.5	27.51
	0.25	400	400	350	300	7.5	26.19
	0.5	450	400	350	300	8.0	20.01
	1	500	450	450	400	8.0	25.01
Commercial sample	0.1	500	450	450	400	7.5	25.98
CD-1	0.25	550	500	450	400	7.5	25.41
	0.5	600	550	500	450	8.0	24.63
	1	650	600	600	550	8.0	24.33

polymer is easy and feasible as it has higher acid value. So we can convert this polymer into water soluble product by neutralization with various alkalies. The HLB ratio point out the possibility of using this novel polymer in liquid, powder and cake detergents

– Various powder detergent compositions based on alkyd polymer give excellent detergency[13] and reduction in surface tension. The foam volume of various composition and foam stability is also good. All these ecofriendly samples have excellent detergency characteristics comparable to commercial sample.

– A synergetic combination of alpha olefin sulphonate and novel alkyd resin give extraordinary result in reference to surface tension reduction, detergency, stain removing and foaming properties. A 50:50 combination of alpha olefin sulphonate and alkyd polymer give optimum result sample P-4 and P-7 shows extra ordinary excellent result. Thus formulation P-4 and P-7 can be recommended for pilot scale studies.

– Sample P-3 and P-8 which is total replacement of acid slurry and alpha olefin sulphonate is also showing appreciable result. A synergetic combination of acid slurry

and novel alkyd polymer give excellent performance characteristics *i.e.* sample P-2 and P-4

– Individual detergency test of alkyd resin at 1 per cent concentration are 83-84 per cent which is comparable or more than individual detergency of commercial active ingredient *i.e.* acid slurry, sodium laurel sulphonate and alpha olefin sulphonate this again shows that our alkyd resin has total replacement of these active ingredients.

– Raw material cost of this detergent powder come around Rs. 28/kg which is certainly attractive proposition. There is an urgent need to take up these composition on pilot plan and commercial scale. Our samples are not only comparable but in some instance superior to commercial samples.

Authors' affiliations:
PRAVIN A. DHAKITE, Department of Applied Chemistry, Shri Shankarprasad Agnihotri College of Engineering, Ramnagar, WARDHA (M.S.) INDIA
B.W. PHATE, Department of Applied Chemistry, Indira Gandhi Priyadarshini College of Engineering, NAGPUR (M.S.) INDIA

REFERENCES

1. **Fulzele, S.V.,** Satturwar, P.M., Gogte, B.B., Dorle, A.K. (). Rosin and its Derivatives pharmaceutical applications, email : fsuniket@yahoo.co.in.
2. **Gogte, B.B.**, Bhagwat, A.M. (2004). *J. Soaps Deter. Toilet Rev.*, **36** : 20-25.
3. Gogte, B.B. and Dontulwar, J.R. (2004). *Asian J. Chem.*, **16** : 1385-1390.
4. **Kirk, O.** (). *Encyclopedia Chemical Technol.*,(11) : 269-270.

   http:/www.nirri.org
5. **Garrett, H.E.** (1972). Surface active chemicals Programmer Press, New York.
6. ASTM standard method 6.01, d1639-70(for acid value of organic coating material), published by the American Society for Testing Material, Philadelphia, 1981.
7. **Jellinia, Stephan** (1982). *J. Encyclopedia of Chemical Technol.*, 20, John Wiley & sons, New York, 1982, p-780
8. **Harris, J.C.** (1984). Detergency Evaluation & Testing Inters Science Publisher, Inc., New York, 1984
9. National institute of Hand Book of industrial Research, Hand Book of Soaps, Detergent andacids Slurry by N.I.I.R. Board.
10. **Bauvy, A.** and Leversidge, P. (1985). Polymeric Surfactantand their use in industrial Application, Paint india, March-1985, p.45-52.
11. **Kirk, Othmer** (1982). Encyclopedia of chemical Technology, 20, John Willey and Sons, NewYork, 1982, p. 780-805.
12. **Suri, S.K.**, Synthetic detergent powder changing Trends –I, Soaps Detergents and Toiletries Review, September-200,p.14-18.
13. **Kharkate, S.K.,** Kardbhajne, V.Y. and Gogte, B.B. (2005). some resin based Ecofriendly Liquid Detergents , *J. Scientific & Industrial Res.*, **64** : 752-755.

****
******
***