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NOVELRESINBASED LIQUID SURFACTANT FOR CELLULOSIC AND PROTEIN FIBERS

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Abstract: -

Novel polymer based on sorbitol and maleic & phthalic anhydride has been synthesized. The mole ratio standardized to get desire molecular weight, HLB ratio and surfactant properties. Liquid detergents have been formulated based on these novel polymer and conventional active ingredient. This polymer has been used as a total replacement of conventional LABS in detergent formulation. The proportion of SLS and SLES has been varied to know the optimum performance. The percentage detergency along with stain removing characteristic, surface tension, and foam height have been evaluated and compared with commercial samples. This composition is producing excellent result. The different types of stains can be effectively removed from cellulose fibers as well as protein fibers like cotton, linen, wool and silk. The use of novel polymer for developing foamless liquid detergent will be useful in washing machines and in the areas with water scarcity. Some formulations are not only technically excellent but also cost effective as compared to commercial products. It promotes green aspects by substituting petroleum based products, their souring cost and availability.

Key word:- Novel Resin, Alpha Olefin Sulphonate, SLS and SLES, cellulose fibers and protein fibers.

Introduction:-

Its high to replace petroleum based surfactant with renewable alternative product. It has been observed that LABS, the most important surfactant as compared to the others. The environmental pollution mainly foaming river and eutrophication are the major concerns. The idea is to prepare composition with minimum use of conventional material and free from petroleum derivative.

Surfactant based on novel resin have been used in various industrial procedures like wall finishes[1], water thinable paints[2], electrodeposition paints[3], water thinable primers[4] and printing inks[5]. We have already used novel polymeric surfactant for production of powder detergent. Resin based surfactants are an exciting new addition to the existing products. Polymeric surfactants when incorporated into detergent they offer features like clay soil disperancy, prevention of soil redeposit ion, fabric anti incrustation, calcium salt inhibition and Ca and Mg sequerterization.

The special features of novel resin based liquid detergent are

1. Excellent stain removing capacity.
2. Complete water solubility.
3. Prevention of soil re-deposition.
4. Free from phosphate.

In our laboratory we have successfully used vegetable stock based polymeric surfactants as replacement of acid slurry to the extent of 50-60%. In the present work experimental conditions have been workout for getting a novel polymer based mainly on sorbitol and combination of maleic anhydride, phthalic anhydride. The experimental additions have been set up to get desired molecular weight, HLB ratio and detergency characteristics. Sodium bisulphate and sodium bisulphite used as catalyst, benzoic acid is used as a chain terminator.

The synthesized polymers were analyzed systematically for their acid value[7], saponification value[8], surface tension[9] and other Physico-chemical characteristics. These products compete in price and technical performance with present generation commercial products.

Experimental:

The Reactor: The preparation of resin was carried out in glass reactor. The reactor consists of two parts. Lower part of the reactor is a round bottom vessel with very wide mouth. The upper part of the reactor is its lid, having four necks with standard joints.

A major 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 an electric heating mantle having a special arrangement for smooth control of the temperature of the reactor. A regulator controlled the speed of the stirrer. The reaction vessel and its lid were tried together with the help of clamp.

Preparation of Novel Resin: -The polymer was prepared in an above reactor. The composition of novel resin mainly based on Sorbitol, Maleic and phthalic anhydride. Sodium Bisulphate and sodium Biulphite has been used as catalyst and Benzoic acid used as chain stopper.

The preparation and cooking schedule of Novel resin is given in Table no. 1 and 2.

Neutralization of Novel Resin:100 gm of Novel polymer was heated to 70℃ the calculated amount of 30% KOH was added to novel polymer with constant stirring so as get slightly alkaline solution of polymer with pH of 8.

Physicochemical Analysis of powder Detergent:

Stain preparation

The soil medium of following composition was prepared.

Component (Weight %) -carbon black (28.4%), coconut oil (35.8%), Lauric acid (17.9%), mineral oil (17.9%). The mixture of carbon black and Lauric acid along with mineral oil was taken in a pastel mortar. Coconut oil was added slowly to form a thick paste. All the components were ground in pastel for 1-2 hours to obtain fine paste.

Soil solution

This was prepared by adding 2gms of above paste in 500ml of carbon tetrachloride. Mix it well and use for staining cloth sample preparation. The solution was kept in packed bottles.

Tea stain solution

The tea was prepared with following composition. Tea (Parivar) (2.2%), sugar (8.0%), milk (38.4%), water (51.4%).

25gms of water was warmed to 35 to 40℃ then add Parivar brand tea and sugar heated up to 80℃ milk was added then heating continued at boil for next 5 minutes, stop heating and pass the tea through a Steiner. Use this as tea medium.

Preparation of coffee medium

The Coffee of following composition was prepared, coffee (1.0%), sugar (8.1%), milk (51.9%), water (39.0%), 25gms of milk and water in a beaker warmed to 35-40℃, coffee and sugar were added and heating continued to the boil for 5 minutes.

Method of application of soil

The cloth of size 24 × 32 cm^2 were prepared. Took 50ml of soil solution in a beaker, the cloth sample was soaked in for 5 minutes. This is kept outside for drying in open atmosphere for 2 hours. Then this cloth was cut in size of 6× 8 cm^2 and samples were used for the washing test.

Method of washing

The solution of different concentration were prepared. Heated to 60℃. Soiled cloth sample was dipped in it for 5 minutes and given to and fro 10 hand washes.

Method of application of (Tea and coffee)

The cotton cloths of size 24 × 32 cm^2 were taken and drawn checks of size 6× 8 cm^2. Then took the above staining solution in a pipette and added in a center of checks one drop and then kept the stain cloth sample in an oven at 55-60℃ for ½ hr. then this stain cloth was cut into test sample size and these were used for stain removal testing. Same method is used for washing.

After washing, the percent detergency[4] 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 reflection photometer with filter R-46 against an MgO-standard.

Table 1. Composition of Novel Resin.

Serial No.	Raw materials	Concentration (% by weight)	
		A	B
1.	Sorbitol (70% solid)	77.42	98.04
2.	Maleic anhydride	10.05	-
3.	Phthalic anhydride	8.00	
4.	Benzoic acid	2.57	2.57
5.	Sodium bisulphate	1.47	1.47
6.	Sodium bisuphite	0.49	0.49

Table 2. Heating Schedule of Novel resin:

Procedure : Total Charge – 2Kg

Sorbitol +Maleic Anhydride + Phthalic Anhydride + Benzoic acid + Sodium bisulphate + Sodium bisulphite

⇩

Mixed all ingredients add to the reactor

⇩

Heat at 120℃ for 1 Hrs

⇩

Heat at 130 ℃ for 1 Hrs and 30 minutes

⇩

Add 100 ml water to maintain paste like consistency

⇩

Heat at 150 ℃ for 3 Hrs

⇩

Maintain temperature for another 30 minutes

⇩

Cool at 60℃

⇩

Thin down the resin with water.

Total time of Heating: 5 Hrs and 30 minutes

Table 3. Physicochemical properties of the synthesized polymers

Sr.no.	properties	Analysis result
1	Solubility	Alcohol & water
2	% solid	93.34
3	Acid value	122
4	Saponification value	255.94
5	HLB Ratio	15.10
6	pH	1.28
7	Colour	Brownish
8	Molecular weight	2,653
9	Viscosity in ethyl alcohol(70:30)	180 second

Table 4. Composition of liquid detergent based on novel resin and SLS.

Serial No.	Raw materials	Concentration (% by weight)				
		LD1	LD2	LD3	LD4	LD5
1.	SLS	10	10	5	2	2
2.	SLES	4.5	4.5	4.5	4.5	3
3.	Resin	---	3.5	7	9.1	12.6
4	Sodium sulphate	5	5	5	5	5
5	Foam booster	1	1	1	1	1
6	Sorbitol	7	7	7	7	7
7	Alpha olefin sulphonate	7	3.5	3.5	3.5	3.5
8	Moisture content	65.5	65.5	67.0	67.9	65.9

Table 5. Analysis of liquid detergents at 0.25% concentration by weight

Detergent sample	Density (g/cc)	Surface Tension(dynes/cm)	PH	Foam height (cc)	% Reduction in surface tension	Viscosity in second
LD1	0.9926	24.26	8.36	405	66.33	279
LD2	0.9917	24.34	7.26	354	65.98	220
LD3	0.9923	24.57	7.36	351	66.02	164
LD4	0.9941	25.61	7.24	290	65.21	103
LD5	0.9927	24.72	7.48	207	66.56	82
Commercial sample	0.8920	21.97	9.05	280	69.55	90

Table No. 6 Effect of detergents based on novel resin on % detergency

Cloth	**Medium Staining**	**Conc.**	**% Detergency**						**Comm. Sample I**	**Comm. Sample II**
			LD1	**LD2**	**LD3**	**LD4**	**LD5**	**LD6**		
Cotton	Soil solution	0.1	86.91	84.73	87.35	89.09	95.63	91.27	83.39	80.13
		0.25	87.35	87.35	88.44	91.71	96.07	93.67	83.39	85.34
		0.5	89.09	91.27	89.53	93.89	96.94	94.76	87.95	85.64
		1.0	90.40	93.89	91.27	95.20	97.81	95.63	97.07	93.48
	Tea solution	0.1	82.79	84.40	86.55	87.90	81.18	81.18	83.06	89.78
		0.25	83.87	87.09	89.51	89.51	90.86	84.40	83.06	86.28
		0.5	87.09	89.24	91.93	91.93	91.93	87.36	77.68	78.22
		1.0	88.70	91.39	93.27	92.74	94.89	89.24	84.94	80.91
	Coffee solution	0.1	88.23	91.39	91.93	92.74	90.86	87.09	93.82	86.76
		0.25	92.20	91.39	92.74	93.54	93.81	89.24	98.23	87.94
		0.5	93.54	91.93	94.62	95.16	95.16	90.05	99.70	91.76
		1.0	94.62	94.89	95.43	95.43	95.43	91.93	97.35	90.20
Linen	Soil solution	0.1	84.23	86.17	88.65	90.33	87.59	88.65	93.11	80.86
		0.25	85.70	88.65	90.33	91.59	88.23	90.75	91.33	88.27
		0.5	86.12	90.33	91.59	92.43	92.85	92.64	94.39	93.36
		1.0	88.23	92.64	92.43	93.69	94.53	93.90	97.70	92.34
	Tea solution	0.1	78.76	84.67	87.09	84.13	81.18	78.49	76.82	93.90
		0.25	81.18	86.56	89.24	89.24	84.49	83.87	90.65	98.78
		0.5	84.13	89.78	91.93	90.86	87.36	86.56	99.18	95.33
		1.0	86.56	91.93	92.47	91.93	89.24	88.70	74.39	100.00
	Coffee solution	0.1	80.45	83.63	84.54	88.18	80.00	79.09	96.90	93.63
		0.25	81.81	85.00	86.36	88.63	82.72	80.00	96.90	66.81
		0.5	83.63	88.18	87.27	88.63	84.54	83.63	96.46	70.18
		1.0	86.36	89.09	89.09	89.09	88.18	85.00	96.46	76.18
Wool	Soil solution	0.1	81.05	85.00	82.64	85.00	86.74	82.39	87.14	78.74
		0.25	83.70	86.74	85.00	86.74	86.74	84.78	88.71	80.31
		0.5	85.00	89.35	86.74	88.04	89.35	86.74	85.83	85.56
		1.0	86.74	90.22	87.39	89.13	90.22	89.35	74.54	87.40
	Tea solution	0.1	71.61	81.84	79.28	79.28	77.23	76.72	82.09	90.03
		0.25	74.68	82.35	84.29	82.35	79.28	79.53	82.39	95.64
		0.5	78.26	84.39	87.21	84.39	80.81	84.39	94.37	94.85
		1.0	79.79	85.93	87.72	85.72	81.84	85.16	93.09	94.35
	Coffee solution	0.1	85.93	87.46	89.51	88.49	85.93	82.35	93.23	89.24
		0.25	86.95	89.51	90.53	89.51	87.46	85.16	93.23	87.64
		0.5	88.49	89.76	92.07	90.02	88.49	86.96	91.23	94.02
		1.0	88.49	90.28	92.58	90.28	90.02	89.59	80.44	91.63
Silk	Soil solution	0.1	82.45	83.10	81.64	84.72	86.52	81.79	84.34	87.74
		0.25	82.70	86.74	85.00	85.74	86.74	84.78	88.71	83.34
		0.5	83.00	89.35	86.74	88.24	89.35	86.74	85.12	84.56

		1.0	83.14	90.22	87.39	89.54	89.22	89.35	84.54	86.48
	Tea solution	0.1	72.11	81.84	79.28	79.28	77.23	76.72	79.19	75.03
		0.25	72.68	82.35	84.29	82.35	79.28	79.53	81.89	84.64
		0.5	74.36	84.39	87.21	84.39	80.81	84.39	84.37	82.85
		1.0	78.65	85.93	87.72	85.72	81.84	85.16	93.09	84.35
	Coffee solution	0.1	84.93	84.46	87.55	88.49	84.78	82.35	89.23	86.57
		0.25	86.40	86.51	90.53	89.51	87.32	86.16	90.23	86.24
		0.5	86.89	87.76	92.07	90.47	88.69	86.96	91.23	84.75
		1.0	87.69	89.28	91.58	90.28	90.02	89.59	80.44	86.53

Result and Discussion:

Novel resin polymeric composition **Table 1** and flow chart of preparation **Table 2** is given. The amount of sorbitol in resin was quite high(78%) the amount of maleic anhydride was maintain 10% and that of phthalic anhydride about 8% . The analysis of resin is given in **Table3.** The **s**election is based on achieving the desired molecular weight, HLB Ratio, viscosity and acid value.

The formulated liquid detergent the percentage of SLES is constant except in LD5. While the proportion of novel resin and SLS is varied. Sorbitol gives a smooth feel, transparency and clarity to our liquid detergents. **Table 4 & 5** showed the analysis of the formulated liquid detergent polymeric surfactants give excellent characteristics of foaming, surface tension and cleaning stain of soil tea and coffee. % detergency on different fibers compared with standard commercial sample in **Table 6** shows excellent percentage detergency. The % detergency obtain for samples is comparable with commercial liquid sample.

The samples LD1 to LD5 have been studied at slightly varying pH from 7.48 to 8.36 we observed that percentage reduction in surface tension is quite good as compared to the commercial sample at pH 8.5 but percentage detergency is slightly low as compared to commercial sample.

The difference in percentage detergency between cellulosic and protein fiber samples are compared and found that the plant based fibers can be cleaned effectively,but between animal origin fibers silk has more percentage detergency than wool.

The use of sodium tripolyphosphate has also been brought down to minimum. In conventional sample the STPPpercentage is between 20 to 30%. This is certainly on effort to develop phosphate free eco- friendly detergent. All raw materials are of vegetable in origin hence we claim that our formulation of detergents are ecofriendly as compared to commercial one, which still use Higher proportion of linear Alkyl Benzene sulphonate. All ingredients are ecofriendly and have vegetable origin. We have avoided the use of petroleum based acid slurry.

CONCLUSION:

Novel resin based on liquid surfactant has been synthesize successfully used as a replacement for petroleum based LABS. The polymer successfully used in liquid detergent composition. There is global trend towards the use of liquid detergent.The use of pollution causing chemicals like STPP is reduced greatly. With this green eco-friendly renewable product, it is a step towards replacement of petroleum product.The liquid detergent formulation in association with SLS instead of using LABS polymeric surfactants has demonstrated their utility as an active ingredient of detergent.

Our sample have been compared in all respects with commercial sample available in the market. Our sample stand up to the mark and sometimes perform better than commercial sample.The present research work will open up a new horizon of using novel resin polymers in surfactant.

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