FOAMLESS POWDER DETERGENT BASED ON NOVEL POLYMERIC SURFACTANS

Dr. N. H. Chahande[1] and Mr. S.S. Pohare[2]

Department of Chemistry, VPMK's Arts, Commerce and Science College Kinhavali, Talula: shahapur Dist: Thane-421404, India.

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Abstract: -

The foamless detergents are required in specially areas such as washing machine, floor cleanser and Toilet cleaning preparations. In this study efforts were made to prepare foamless detergent by using polymeric surfactant (Novel Polymer). Novel eco-friendly polymer based on sorbitol and Maleic anhydride has been synthesized. The mole ratio of ingredients and cooking schedule has been standardized to get desire molecular weight, H.L.B. ratio, PH and surfactant properties. This polymer has been used as a total replacement of conventional LABS in detergent powder formulations. The proportion of dolomite has been varied to know the optimum performance. The surface tension, foam height, % detergency and stain removing characteristics have been evaluated and compared with commercial powder detergent samples. Some formulations which are technically excellent and yet cost effective have been identified for commercial production.

Introduction.

The relationship between foaming power and detergency has always been of interest, and foaming power has become associated in many consumers minds with high detergent power it is generally connected by detergent technologists that foam height has no direct relationship to cleaning power in ordinary fabric washing systems.

In certain detergent operations high foam is a definite requirement, in certain cases it is immaterial whether the detergent foam or not, and in other cases foam can be considered a nuistance. It is obvious that a hair shampoo, shaving cream (other than the brushless type) and bubble bath preparations need to produce copious foam.

Commercial and automatic house-hold laundry machines are almost without exception foam sensitive. If the detergent foams unduly the foam overflows on to the floor and also can interfere with the free flow of clothes through the water. In automatic machines the foam can interfere with water level pumps and the proper working of the controls in the machine. A small amount of foam is necessary as this tends to trap dirt particles.

The foamless detergent will be boon to water scarcity areas. There are water scarcity areas in Rajasthan where nobody can afford more water for cleaning. There is definite water saving using our foamless detergents. The present work is an attempt to develop some foamless formulations based on sorbitol and maleic anhydride based novel resin.

This is a global need to replace petroleum based raw materials with eco-friendly renewable green materials. The incorporation of polymeric surfactant has positive effect on the following effect on the following performance features of detergents viz

- As a substitutes for Phosphates in detergent.
- As a soil release agent in detergent
- As a anti redeposition agent in detergent.
- As an emulsifier in Meta-stable emulsion for cold rolling of steel.
- As a compatibilizers in immiscible polymer blends

- Polymeric surfactant in non-aqueous system (Dispersion polymerization)

In the present research work experimental conditions have been worked out for getting a novel resin based mainly on sorbitol and maleic anhydride. The experimental conditions have been set up to get desired molecular weight, HLB ratio, and desired characteristics. In powder detergent formulations very small quantities of STPP have been used.

The special feature of our formulations is used of 15 to 40% dolomite in detergents powder dolomite gives whiteness and brightness to cloths and many commercial formulations it is reported, therefore special efforts have been undertaken to understand the role of dolomite in detergent composition.

The effect of dolomite on various parameters like surface tension, foam height, moisture content, and alcohol stability are studied. Finally, these detergent powder samples are analysed for cleaning action on various types of cloths such as terricot, cotton and polyester.

The basic idea of this research work to get detergent composition, having excellent functional properties like cleaning, foaming, wetting emulsifying, solubilising and dispersing in cheapest rate.

Experimental:

A glass reactor fitted with stirrer, heating mantle and condenser has been used in the synthesis of novel polymers. The temperature control of 2^0C can be achieved by using an efficient temperature regulator. A constant water supply through a condenser helps to control reactor temperature. Initially stoichiometric quantity of sorbitol, Maleic anhydride and benzoic acid were added in the reactor. Sodium bisulphate and sodium bisulphite were used as a catalyst. The temperature was raised slowly and steadily in about 0.5 hr. to 150^0C. The reaction was continued for 5 hr. and 30 min. till the desired molecular weight was achieved. The consistency of paste was maintained by adding 2% water as a solvent. At the end of this period the reaction was terminated and prepared polymer was collected in a glass stoppered bottle with least air gap. The final yield of the product was measured.

Table No. 1
Composition of Novel Resin

Ingredients	% By Weight
Sorbitol	77.42
Maleic Anhydride	18.05
Benzoic Acid	2.57
Sodium bisulphite	0.49
Sodium bisulphate	1.47

In this formulation 2% solvent (water) was added on weight basis. Total heating hrs = 5.30 min.
Temperature range = 120^0C to 150^0C

Table No. 2 Analysis of Novel Resin

Sr. No.	Properties	Analysis Result
1	Solubility	Alcohol and 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	Reddish brown
8	Molecular weight	2,653
9	Viscosity (70:30) in ethyl alcohol (By ford cup method)	180 sec.

Preparation of Powder detergents: -

Five different compositions of powder detergents have been prepared based mainly on neutralized novel polymer, sodium carbonate, urea, STPP (Sodium tripolyphosphate), CMC (Carboxymethyl cellulose), Sorbitol, SLS (Sodium lauryl sulphate), and dolomite etc. STPP has been maintained at lower level of 2-3% in this method neutralized novel polymer sodium bicarbonate, sodium lauryl sulphate, dolomite, CMC etc. according to compositions were taken in a blender. All the contents were mixed together for 30 minutes in a powder blender. After ensuring the intimate contact of all the contents in a mixture the detergents were taken out. The powder detergents were dried to get required moisture. After getting dry, free flowing property. The sample were packed into air tight polyethylene bags.

Table No. 3 composition of powder detergents

Sr. No.	Ingredients	PD1	PD2	PD3	PD4	PD5
1	Sodium carbonate	38.22	35.24	31.26	27.73	24.11
2	Urea	6.94	6.40	5.62	5.04	4.38
3	Sodium Sulphate	6.94	6.40	5.62	5.04	4.38
4	STPP	3.47	3.19	2.84	2.51	2.18
5	CMC	0.27	0.25	0.23	0.20	0.17
6	Sorbitol (100% solid)	2.07	1.91	1.70	1.51	1.31
7	SLS (100% solid)	2.07	1.91	1.70	1.51	1.31
8	Neutralized Novel Resin (100% solid)	11.95	11.19	9.94	8.83	7.66
9	Dolomite	8.02	16.64	25.29	34.91	45.51
10	Moisture content	20.05	16.87	15.80	12.72	8.99

(Neutralized Novel polymer was used as 70% solid)

Analysis and Testing of powder detergents

Surface Tension: The surface tension of powder detergent was measured using stalagmometer.

Foam Volume: Foam is 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 characteristics were measured in terms of volume by Bubble Cylinder Method.

Detergency Test: This includes the following steps:

Preparation of soil medium: 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.
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.

Fabrics washing: The solutions of 0.1%, 0.25%, 0.5%. 1.0% concentration of powder detergents in tap water were prepared. These solutions were heated to 60^0C and stained fabrics were dipped in it for five minutes. Ten to and fro handwashes 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 washing, the percent detergency was found out by using Lambert and Sanders Formula.

$$\% \text{ Detergency} = \frac{(Rw - Rs \times 100}{R0 - Rs}$$

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 and MgO-standard.

Table No. 4 Analysis of powder Detergents

Conc.	Sample	Foam volume in Time in min.				Density g/ Cm^3	Surface Tension Dyne/Cm^3	% Reduction in Surface Tension
0.1%		**0**	**5**	**10**	**15**			
	PD1	30	20	20	10	0.9944	52.68	25.99
	PD2	20	20	20	20	0.9970	54.71	23.14
	PD3	20	20	10	10	0.9988	56.83	20.60
	PD4	20	10	10	--	1.0048	65.69	7.71
	PD5	20	10	--	--	1.0055	68.66	3.54
	Comm. Det.	280	250	200	180	0.9875	29.59	58.89
0.25%								
	PD1	20	20	10	10	1.0003	49.57	30.35
	PD2	20	10	10	10	1.0014	52.16	26.72
	PD3	20	10	10	10	1.0003	57.10	19.78
	PD4	20	20	10	--	1.0059	60.61	14.85
	PD5	20	20	--	--	1.0070	64.46	9.44
	Comm. Det.	400	280	210	180	0.9900	26.52	63.15
0.5%								
	PD1	20	10	10	10	1.0007	45.89	35.52
	PD2	20	20	10	--	1.0048	48.25	32.21
	PD3	20	10	10	10	1.0059	50.67	28.81
	PD4	20	10	10	10	1.0074	52.47	26.28
	PD5	20	20	10	--	1.0081	56.32	20.88
	Comm. Det.	550	420	260	220	0.9945	24.14	66.46
1.0%								
	PD1	30	20	20	20	1.0066	43.56	38.80
	PD2	30	20	10	10	1.0074	44.87	36.96
	PD3	30	20	10	10	1.0081	46.23	35.05
	PD4	20	20	20	20	1.0010	48.50	31.86
	PD5	20	10	10	10	1.0011	52.67	26.01
	Comm. Det.	680	420	380	270	0.9996	22.84	68.26

Table No. 6
Soil, Tea, Coffee Stain on terricot, Cotton, Polyester Fabric % Detergency

Reflectance on clean Polyester cloth R0 = 72.7 Reflectance on soil, Tea, Coffee-Stained Polyester Cloth R= 48.8
Reflectance on clean Terricot cloth R0 = 74.2 Reflectance on soil, Tea, Coffee-Stained Terricot Cloth R= 41.3
Reflectance on clean Cotton cloth R0 = 72.1 Reflectance on soil, Tea, Coffee-Stained Cotton Cloth R= 40.3

Cloth	Medium for staining	Conc.	% Detergency of powder detergent sample					Comm. Sample 1	Comm. Sample 2
			PD1	PD2	PD3	PD4	PD5		
POLYESTER	**Soil solution**	0.1%	75.51	75.25	72.19	66.83	77.29	93.11	80.13
		025%	69.89	77.29	72.95	66.07	76.78	91.33	80.34
		05%	59.54	75.51	72.17	73.21	73.21	94.39	80.64
		1.0%	69.64	72.19	78.82	73.21	63.01	97.70	93.48
	Tea solution	0.1%	77.23	61.38	73.98	51.21	50.40	76.82	89.78
		025%	68.29	80.48	60.16	51.62	39.02	9.65	86.28
		05%	76.42	53.65	67.07	50.00	42.68	99.19	78.22
		1.0%	60.16	60.97	65.85	59.34	61.78	74.39	80.91
	Coffee solution	0.1%	80.00	95.90	85.90	94.54	88.18	96.90	86.76
		025%	90.90	90.09	87.27	93.63	95.91	96.90	87.94
		05%	96.36	97.27	91.81	91.81	94.06	96.46	91.76
		1.0%	96.00	95.06	95.00	95.45	96.13	96.48	90.20
TERRICOT	**Soil solution**	0.1%	67.19	80.06	60.10	80.06	82.20	97.14	80.86
		025%	71.65	82.40	73.49	70.80	64.82	88.71	88.27
		05%	76.90	69.55	58.79	64.56	62.72	85.83	93.36
		1.0%	81.88	73.75	74.01	70.60	65.09	74.54	92.34
	Tea solution	0.1%	77.23	79.79	86.44	70.33	87.21	82.09	93.90
		025%	96.67	93.35	95.90	93.60	85.93	82.39	98.78
		05%	95.14	99.76	97.18	94.88	95.65	94.37	95.33
		1.0%	98.46	96.41	90.53	97.44	95.90	93.09	100.0
	Coffee solution	0.1%	86.05	85.65	84.06	90.83	86.83	93.23	93.63
		025%	93.62	82.26	86.06	90.03	83.66	93.23	66.81
		05%	91.63	90.83	80.87	90.83	89.64	90.44	70.18
		1.0%	92.82	88.04	81.67	90.43	86.65	83.31	76.18
COTTON	**Soil solution**	0.1%	81.43	78.50	76.54	87.62	90.83	83.39	78.74
		025%	69.70	85.99	67.75	83.06	90.18	82.39	80.81
		05%	76.22	76.87	79.80	79.47	55.04	87.95	85.56
		1.0%	77.19	77.52	85.01	80.45	72.63	97.07	87.40
	Tea solution	0.1%	78.49	81.72	84.13	77.15	72.13	78.76	90.03
		025%	78.76	75.80	84.13	83.06	87.90	83.06	95.64
		05%	84.40	82.52	83.87	71.77	71.77	77.68	94.85
		1.0%	83.87	76.07	77.15	78.76	69.08	84.94	94.35
	Coffee solution	0.1%	92.94	93.52	81.76	87.94	87.35	93.82	89.24
		025%	100.02	89.49	83.23	89.41	80.29	98.23	87.64
		05%	97.35	90.29	80.29	85.88	88.52	99.70	94.02
		1.0%	90.88	92.64	82.35	88.82	95.88	97.35	91.63

Result and Discussion:

Carbohydrate Polymers have become very important because of the demand of Biodegradable Polymers. In our country vegetable-based polyol like sorbitol is abundantly available and cheap. Our detergent industry is totally dependent on petroleum-based products like linear alky! Benzene sulphonates so in this research work we have tried to develop polymeric surfactant based on sorbitol and maleic anhydride. The selection is based on achieving desired molecular weight, H.L.B Ratio, viscosity and acid value. The analysis of resin is given in Table No. 2. The composition of various detergent powders based on this Novel resin are given in Table No. 3

The proportion of neutralized resin has been progressively increased from 7.66 to 11.95. A small proportion of sodium lauryl sulphate has been incorporated. (1.31 to 2.07%). The speciality of formulations is lower % of Sodium Tripolyphosphate about 2 to 4% thus the formulations are eco-friendly. A small proportion of sorbitol gives a smooth feel to detergent.

In common household detergents Dolomite (Mg & Ca Carbonate & Silicate) is a common ingredient in low-cost detergents. The present formulations are carried out with a view to know the positive effect of dolomite on various parameters of detergents like whiteness, detergency, surface tension & foam height. In our earlier experiments we observed that Dolomite has positive effects on many properties. In successive formulations PD1 to PD5 the proportions of dolomite have been increased from 8.2 to 45.0 %. Dolomite has been used by substituting mainly Sodium carbonate, this will reduce the cost of detergents. The cost of Sodium carbonate is around Rs. 15/- per kg. while white grade of Dolomite is Rs. 5/- per kg.

Table No.3 gives compositions of dolomite-based powder detergents. The Overall percentage of active materials has been maintained at a lower level 7.66 to 11.95.%. The idea is to know how Dolomite is effective at lower concentration, of active ingredients. The % Solids of these formulations is varying from 81 to 91.01, %. Slightly higher % of moisture is present compared to commercial sample. The % reduction in surface tension is up to 30 %. This is inferior to commercial samples and needs some improvement table No. 4.

Samples prepared are low foaming as compared to conventional commercial sample. Foaming characteristic are not up to the mark However these results indicated that we can promote these sample as foamless detergent as new concept of foamless detergent Coming into the market for saving of water.

All these samples based on Dolomite are foamless detergents so they can be used in washing machine, Floor cleansers &other foamless industrial detergents. The % detergency on soiled stained cloth is excellent table No. 5.

The sample PD2 containing 20 % Dolomite gives highest detergency comparable to commercial samples. All types of stained cloths (cotton, polyester & terricot) show excellent detergency at 0.25 % conc. This is certainly an important finding it is possible to device a formulation which is effective at a very low conc. of active materials (7.66 to 11.95 %) with cheaper substitution of Dolomite. This preparation is foamless which is again a special feature.

The stain removing properties for coffee is excellent. More than 90 % detergency in stain-removed cloth is reported. The stain removing is equally good for any type of cloth polyester terricot & cotton. Thus, there appears a positive role of Dolomite in stain removal of various types of cloths and various types of stains.

Conclusion:

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

1. Novel Polymer based on Sorbitol & maleic Anhydride can be used with advantage. for formulations of powder detergents. Synergesic combinations along with Sodium Lauryl Sulphate can be worked out which give excellent % reductions in surface tension & distinct stain removing properties for soil & coffee.
2. 7.66 to 11.95 % Neutralized Novel Polymer has been used in different synergesic combinations successfully.
3. Samples PD1 to PD5 are foamless but still they give excellent reduction-in surface tension, soil removal & stain removing characteristics. They can be used for special. applications like washing machine, floor cleansers & industrial surfactants without foam.
4. Dolomite has been substituted for sodium carbonate in successive compositions (8.02 to.45.51%). The proportion of Neutralized Novel polymer is varied from 7.66 to 11.95 %. The amount of Sodium Lauryl Sulphate is also low. The synergesic combinations of Neutralized Novel resin & Dolomite give excellent soil removal & stair removal characteristics. These can be promoted as foamless detergents.
5. Use of 10-20 % Dolomite gives excellent detergency characteristics and % reduction in surface tension. (Samples PD1, PD2)

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