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SYNTHESIS AND STUDY OF CLEANING CHARACTERISTICS OF SORBITOL – STARCH BASED POLYMERIC SURFACTANTS

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Abstract: Polymeric surfactant based on sorbitol and starch has been successfully synthesized. In the present piece of research work major quantity of sorbitol and maize starch and lower quantity of organic acid and anhydride is used for the synthesis of polymeric surfactants. These polymers may be ecofriendly as they are totally vegetable based product. The HLB ratio of polymers clearly indicates it's used in detergents. All these polymers has a potential to substitute petroleum based acid slurry and alpha olefin sulfonate. Preparation of polymers is comparable and suitable for commercial production. Economically they are cheaper than petroleum based product.

Key Words: **- Sorbitol, Starch, organic acids, polymeric surfactant.**

I. INTRODUCTION

The most important use of polymeric surfactant [1] is for making detergents. The Special polymeric surfactants are added to a detergent formulation as soil-release agents that modify fabrics surface to make it easier to clean. Generally most of the commercial detergents contain petroleum based product like acid slurry and alpha olefin sulphonate (AOS). These chemicals have created a big threat to environment due to their tendency of non-biodegradable. These conventional products are non-renewable, highly expensive band exhausting the available natural resources. Another important commonly used binder in detergent system is sodium tripolyphpsphate (STPP). There is growing environmental consequences against STPP. All these three components viz., Acid slurry, AOS and STPP are not ecofriendly. There is a need to replace it or decrease it's concentration in detergents. We must developed renewable vegetable based alternatives for these conventional petroleum based surfactants.

The present research work is aimed to develop a polymeric surfactant based upon sorbitol and maize starch. It will have certain characteristics and it is used in the detergent formulation as partial or total substitute for Acid Slurry and alpha olefin sulphonate.

II. EXPERIMENTAL

The Reactor for Synthesis:

The reactor used in synthesis of polymeric surfactant is made up of glass. It has two parts. Lower part is a round bottom flask while upper part is its lid having four opening neck with standard joint. Generally central opening is for mechanical stirrer to mix the reaction mixture. Out of remaining three opening, one is for adding raw material, second is for connecting the water condenser and third is for putting thermometer. The reaction mixture is heated with the use of electric heating mantle. Mechanical stirrer with speed regulator is used to stir the reaction mixture.

Synthesis of Polymeric Surfactants:

In this type of polymers sorbirol and starch (maize) were main ingredients. We have heat it with different proportion of organic acids at 130^0C for three hours. Total Four batches were prepared. The percentage of ingredients in different compositions is given in table 1.

Table 1: Percent Composition by weight of polymeric surfactants

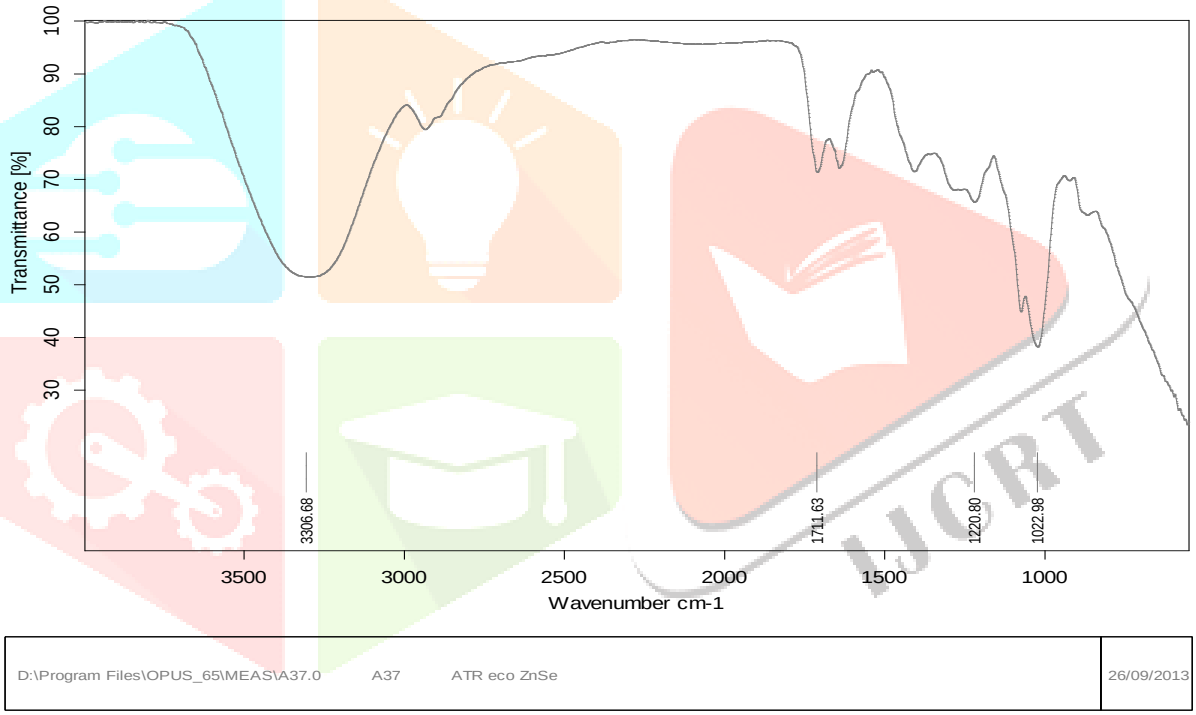
Ingredients	A_{37}	A_{40}	A_{43}	A_{44}
Sorbitol (70%)	49	48.5	57.14	47.5
Starch (70%)	39.1	39.4	19.05	28.6
Maleic Anhydride	4.9	4.8	9.52	9.5
Pthaleic Anhydride	2.5	2.4	3.81	6.6
Oxalic Acid	-	-	2.86	-
Citric Acid	2.5	2.4	2.86	2.8
Sodium bisulphate	1.5	-	-	-
Sodium Bisulphite	0.5	-	-	-
Sodium metabisulphite	-	2.5	4.76	5.0

Initially sorbitol, starch maleic anhydride, phthalic anhydride, citric acid, oxalic acid, sodium bisulphate, sodium bisulphite and sodium metabisulphite were taken in a glass reactor. Mass was heated slowly and steadily to 130^0 C for 1.5 hours. This temperature was maintained for three hours. Acid value and viscosity was observed periodically and reaction was terminated when desired acid value and viscosity has attained. Batch was withdrawn carefully and weighted to get % yield.

Physicochemical properties such as % solid [2], viscosity [3], pH, acid value [4], hydrophilic lipophilic balance [5] (H.L.B.), oxirane oxygen value [6], and % detergency [7] and spectroscopic analysis [8-9] (I.R. and Mass) of sorbitol-starch based polymers were studied. The determined properties of polymers are given in table 2 to 5.

Table 2: Physico-chemical Analysis of Polymers

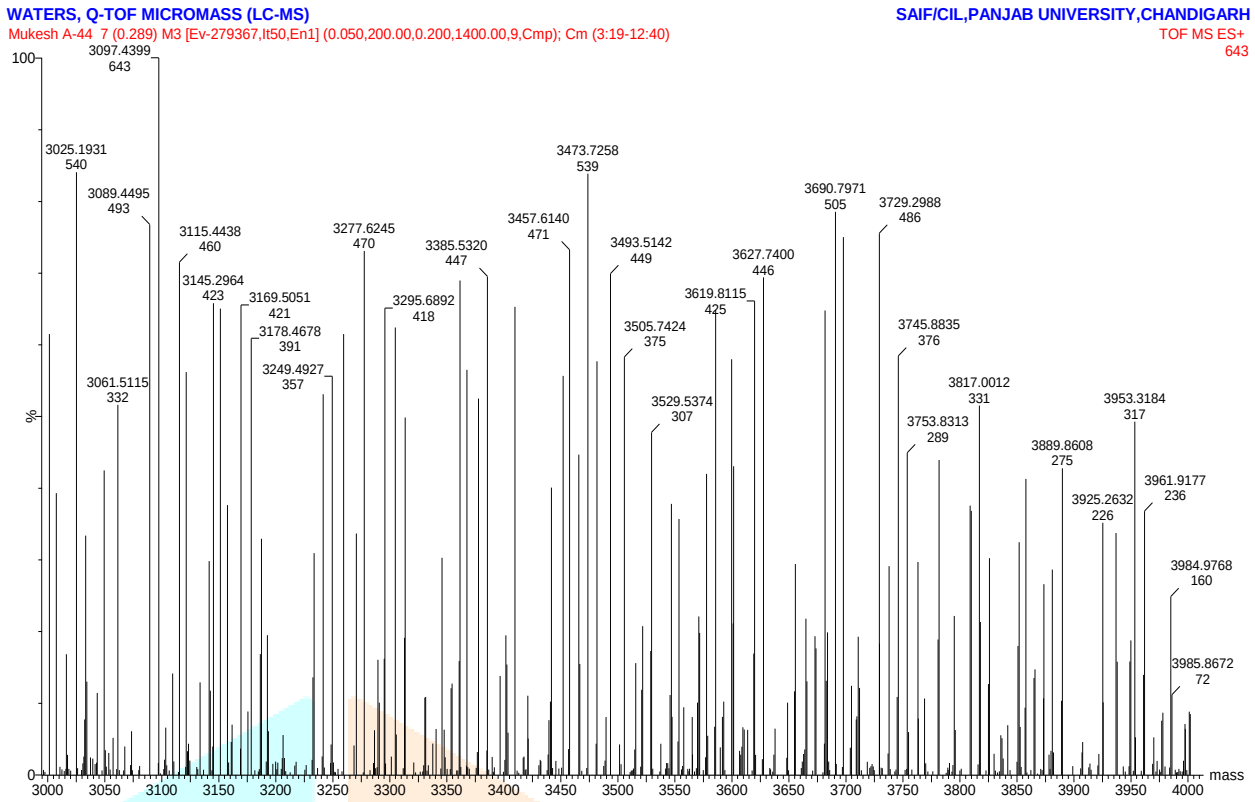
Polymer	% Solid	Viscosity (Sec.)	Colour	pH value	H.L.B. ratio	Oxirane Oxygen value	Surface Tension Dyne/cm	Acid Value
A_{37}	74	183	Yellow	3.76	15.80	0.22	40.50	94.65
A_{40}	77	180	Yellow	4.06	15.38	0.22	65	87.97
A_{43}	77	130	Colourless	3.70	16.42	0.22	68.17	61.2
A_{44}	70.39	180	Reddish Brown	3.76	14.67	0.39	65.20	91.32



Spectra 1: Infra-red spectra of Novel Polymer (A_{37})

Table 3: FT-IR Spectroscopic analysis of Polymer A_{37}

Sr. No	Functional groups	Literature Wave number cm^{-1}	Observed Wave number cm^{-1}
1	Hydroxyl group (OH stretch)	3200-3600	3306.68
2	Ester group C=O	1705-1720	1711.63
3	Ether group (C-O stretching)	1260-1000	1220.80, 1022.96



Spectra 2: Mass spectra of Novel Polymer (A_{44})

Table 4: Stain (Soil and Tea) Removal study with 1 % solution of Sorbitol-Starch based polymers

Batches	Soil		Tea	
	R_W (By reflectance meter)	% Detergency	R_W (By reflectance meter)	% Detergency
A_{37}	82	70.4	85	74.6
A_{40}	79	65.6	81	67.8
A_{43}	77	62.2	79	64.4
A_{44}	80	67.2	82	69.5
Acid Slurry	82	70.4	85	74.6
Sodium Lauryl Ether sulphate	81	68.8	84	72.9

Table 5: Stain (Coffee and Spinach) Removal study with 1 % solution of Sorbitol-Starch based polymers

Batches	Soil		Tea	
	R_W (By reflectance meter)	% Detergency	R_W (By reflectance meter)	% Detergency
A_{37}	83	70.7	88	76.9
A_{40}	81	67.2	83	67.3
A_{43}	80	65.5	81	67.3

A_{44}	82	69.0	85	71.1
Acid Slurry	84	72.4	88	76.9
Sodium Lauryl Ether sulphate	83	70.7	87	75.0

III. RESULT AND DISCUSSION

The compositions of various sorbitol starch based polymers are given in table1. Polyol groups come from sorbitol (70%) solution. The total of carbohydrates used in the entire polymer is 80% in different composition. The ratio of sorbitol to starch has been varied, thus maximum sorbitol is used in A_{43} combination. In our earlier work we have used independently sorbitol and starch to the level of 80%, various organic acid are used were around 20%. This work indicates that we must use combination of sorbitol and starch to get improves results. Therefore in this work a combination sorbitol and starch (Maize) has been used with expectation of improved results. The organic acids used were 20% in which combination of maleic, phthalic citric and oxalic acids has been used. Maleic and phthalic anhydride helps to build molecular weight to the extent of 3000- 4000. Oxalic and citric acid improved capacity of soil and stain removal. In catalyst part we have used either a combination of sodium bisulphate and sodium bisulphite or sodium metabisulphite. In this experiment we have used sodium metabisulphite, so this novel combination can improve the performance of polymer. It was also realized that bisulphite catalyst not only catalyzed reaction but also enter in reaction with hydroxyl group which further help to get surfactant property. Thus all these combination are unique blend of carbohydrates, organic acids and bisulphites. Which are likely to give exciting result, in reference to cleaning stain removing and reduction in surface tension. The prepared polymers were analyzed for various physicochemical properties as given in table 2. The entire polymers content solid contain 70 to 77 %. Appreciable range of viscosity is observed in all the polymers. Normally the samples are colorless to yellow with only exception of polymer A_{44}, which is dark in color. The pH of samples vary from 3-4. The other constants strongly recommend the use of these polymers as partial or total replacement of petroleum based acid slurry. The stain removing property is indicated in table 4 and 5. One percent solution of polymer in water is used for testing. Spinach removal is excellent while stain of tea, coffee and soil are also removed to the desirable extent. Our polymeric cleaning results are comparable with acid slurry and sodium lauryl ether sulphate. This investigation definitely shows that the polymer should be used in powder and liquid detergent composition. Neutralized samples were prepared for IR spectra. IR spectra show presence of hydroxyl, ether and ester functional groups as given in table 3. Mass spectral data of our polymer having the molecular weight in the range of 3000 - 4000.

IV. CONCLUSION

1. Polymer can be synthesized based on vegetable products like sorbitol and starch and to get a partial or total replacement of petroleum based acid slurry or Alpha olefin sulphonate detergent compositions.
2. Fundamentally it is an esterification reaction. The polyhydroxy groups from sorbitol and starch react with various organic acids like maleic, citric or phthalic to form useful surfactant products.
3. Along with esterification, etherification reaction between two internal hydroxyl groups in carbohydrate entity has also been observed. The ether groups will make the polymer hydrophilic.
4. Sodium bisulphate, Sodium bisulphite and sodium metabisulphite acts as a catalyst for esterification. They themselves can react with hydroxyl groups to form sulphonate groups.
5. The mass spectra of polymer indicate that average molecular weights of polymers are in between 3000 to 4000.
6. The mole ratio of reactants, temperature and properties of catalyst can be optimized to get polymer of desired molecular weight and physicochemical properties like surface tension, acid value and cleaning properties.
7. Soil and stain removing properties for all these polymers are good to excellent and comparable to conventional active material like acid slurry.
8. In all composition 19 to 39 % starch has been used. The organic acids used are in between 18-20 %. Sodium bisulphate, sodium bisulphite and sodium metabisulphite used in proportion 2-5%. These chemicals not only catalyzed esterification but also react with hydroxyl group to form sulphonates which has good cleansing and stain removing properties.
9. All samples have percent solid 70 to 77% and reasonable good viscosity. The HLB ratio, pH value and oxyrane oxygen value suggest strongly their used in detergents.
10. Excellent cleaning, soil and stain removing and bright white appearance of cloth are special feature of starch based polymer.

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