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SYNTHESIS OF NOVEL ECOFRIENDLY SUGAR BASED POLYMERS PREPARED BY CONVENTIONAL AND MICROWAVE SYNTHESIS TECHNIQUE

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Abstract- Petroleum materials are the active and useful ingredients in commercial products. However, these products have posed big threat to the environment due their untenable tendencies of non-biodegradable and hazardous nature. Besides, these conventional products are highly expensive, nonrenewable and exhausting the available natural resources. In this context, the replacement of these products with suitable ecofriendly and renewable materials is inevitable. The natural materials and the products derived from natural resources meet all above requirements. To meet these requirements, an idea has explored to develop novel polymers based on natural sources like starch, sorbitol, sugar, glycerol, and small amount of phthalic and maleic anhydride. Latter, these polymers after neutralisation have been incorporated in preparation of liquid detergents formulation, as a partial substitute for LABS so that vegetative origin of products should remain intact. This Novel carbohydrate polymers are easily synthesized by conventional method and by microwave synthesis technique and compared with each other by its method of analysis, and by its application in replacement of petroleum products which are abundantly used in powder and liquid detergent .
Keywords- sugar, starch, sorbitol, synthesis, surfactant.

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Introduction

Today's consumer interest in biodegradable materials has grown as a consequence of increasing social awareness to reduce environmental degradation by selecting more environmental-friendly products. Due to this, globally, the development of polymers and their derived products is a high priority from the standpoint of environmental preservation. In this race, synthetic natural products (like starch, sorbitol, sugar, etc.) based polymers and their allied products are being increasingly explored because of their potential applications.

In the last five decades there is common trend to use petroleum products as active and useful ingredients in commercial products like liquid and powder detergents, paints, printing inks, cosmetics and textile auxiliaries. Commercial detergent powder use LABS in range of 15-20%. There is a need to synthesis novel products based on vegetable origin which are eco-friendly. Efforts are necessary to substitute petroleum based product by novel polymers.

The present generation of surfactant is based on LABS or similar materials, which are obtain from crude petroleum. We must develop renewable vegetative based alternatives for these conventional petroleum based surfactant.

The present research work is aimed at developing a polymeric surfactants based on sorbitol, sugar, maleic anhydride and phathalic anhydride. As the polymeric surfactant is based on sorbitol and sugar it will have certain characteristics. And it is used in the detergent formulation as partial substitute for LABS. We tried to formulate detergent consisting of dolomite, silicate with minimum use of STPP. From the statistical study of production of sugar in India it is observed that surplus amount of sugar is left behind after its supply to the required areas. The cost of sugar is only Rs 35 per kg, so it will be economical and sensible if this excess of remaining sugar is utilized in the synthesis of some commercial products of reasonable price which are required by common man in his day to day life.

Experimental

Polymers from various compositions were prepared by batch process. The steps involved in the preparation of polymer are given below:

1. For preparation of starch-sorbitol polymer, a homogenous dispersion of mixture of sorbitol, maize starch, maleic anhydride, citric acid, oxalic acid, sodium bisulphate and sodium bisulphite was prepared by using electrically controlled homogenizer. Then, dispersion was introduced into specially designed glass reactor.
2. The dispersion mass was slowly heated at 80°C -120°C for 2 hrs.Then mass was slowly cooled and phthalic anhydride and solvent isopropanol were added. Then again heated at 160-225°C for 3 hrs.
3. The reaction mass was observed periodically for its consistency and color. To maintain the flow and homogeneity of mass additional amount of water was added whenever required.
4. Acid value and viscosity were measured periodically and reaction was terminated when sufficient viscosity and acid value had reached. Total water removed was measured. Batch was withdrawn and weighted.

Same procedure was adopted in preparation of sugar-sorbitol polymer. . The prepared polymer was neutralized by adding calculated amount of 30% KOH in hot polymer with constant stirring so that to obtain slightly alkaline solution with pH = 8.

Microwave Heating System

The preparation of resin was carried out in Bench Mate type microwave reactor. The reactor is operated as closed system. Resin from various compositions was prepared by batch process. The reaction temperature and addition of ingredients are detailed below. The sugar, starch sorbitol, maleic anhydride, phathalic anhydride and citric acid are well mixed to give a homogeneous mixture in test tube and test tube is packed with scapta cap. The test tube is introduced into the microwave reactor and then temperature, microwave power and time are adjusted. The mixture was kept for 2 min at 60°C in microwave reactor by setting the time and temperature of reactor. After 2 min the reactor stopped automatically. After cooling, reaction mixture was taken out of the reactor. Five resins of different composition were studied at same temperature and microwave power setting time of 2 min. The sample prepared in microwave batch has slightly lower acid value but other parameters like consistency, HLB ratio and avg. molecular weight are quiet comparable thus the reaction of esterification which takes about 3-5 hours can be completed in just 2 min. The product has cleaning, homogeneous and with good foaming characteristics. The sample has been chosen for formulation of liquid detergent as this has higher and desired HLB ratio and comparatively pink color clarity and molecular weight compared to conventional batch.

Results and Discussion

In the microwave synthesis various carbohydrate polymers based on sugar, maize starch sorbitol and glycerin have been studied. The various parameters like Temperature, Time, and Microwave power have been kept constant. All carbohydrate novel polymers have been analyses for their physicochemical properties like Acid value, color, saponification value, HLB ratio, oxirane oxygen content etc. The carbohydrate polymer (S1) sample has been chosen for preparation of powder and liquid detergent due to good % solids, consistency, color and HLB ratio. To compare the microwave synthesis with conventional reactor synthesis, the sample which is prepared by conventional glass reactor was prepared by microwave reactor . In the conventional method the reaction time was 5 hrs. and temperature 225°c. If we compare (table no.2 comparison between conventional and microwave results) the acid value is slightly higher the results are better than polymer prepared by conventional methods .The carbohydrate polymer prepared by microwave was neutralized by 30%KOH as a conventional method. The polymer was used for the preparation of liquid detergent .The formulations were same as the formulations of liquid detergent prepared by using conventional method. The analysis of liquid detergent shows that the sample prepared by using microwave synthesis are on par and sometime better than conventional heating based polymers. The detergency is also compared with commercial products and the results are better than commercial products.

Conclusions

- The presented result confirmed that the application of microwave heating substantially reduces the reaction time down to several minutes. There is tremendous time saving in microwave synthesis, normally 2-3 hrs. are required for carbohydrate polymer preparation at 225°c the time of reaction is 2 min.
- The following standard conditions can be recommended for synthesis of carbohydrate polymers. Carbohydrate polymers: Time 2 mins, Temp. 60°c and wattage 60.
- The microwave synthesis gives results which are on par or some times better than conventional methods.
- The space required for microwave reactor is very small and man power required can be reduced continuously. The initial investment is higher but the recurring expenses, & energy expenses, are lower in microwave reactor.
- Microwave reactor can be the desired route which is financially and technically viable option for future time.The presented microwave-assisted transesterification method might substitute the toxic, hazardous and time-consuming classical etherification processes in preparing polysaccharide based surfactants.

Table 1- Synthesis of Sugar- Sorbitol based carbohydrate polymers

Ingredients	B1	B3	S1	S4
Sorbitol (70%)	16.5	12	15.6	-
Maleic anhydride	1.5	1.5	1.5	-
Phthalic anhydride	1.5	1.5	0.6	1.5
Starch	7.5	12	3	-
Oxalic acid	1.5	1.5	1.5	1.5
Citric acid	1.5	1.5	1.5	
Sugar (80%)	-	-	6	15
Glycerol	-			12

Note- Sodium bisulphite, Sodium bisulphate, and Isopropanol are used as catalyst.
Batch S1 shows excellent results and hence its comparison is given in table no.2

Table 2- Comparison of Batch S1 (Prepared by conventional and by microwave synthesis method)

Tests	S1(conventional method)	S1(microwave synthesis method)
% Solid	79.52	82.36
Acid value	21.52	20.28
Solubility	Water	Water
Color	Pink	Pink
H.L.B.	15.3	15.7
Molecular weight	5179.78	5234.56
Ester value	126.03	131.87
% Oxirane oxygen value	14.24	15

Table 3- Liquid Detergent composition based on polymer S1 obtained by Microwave synthesis.

Sr. No.	Ingredients	LD1	LD2	LD3	LD4	LD5
1	Acid slurry (70%)	6.0	4.5	3.0	1.5	00
2	AOS	6.0	4.5	3.0	1.5	00
3	SLS	7.5	7.5	7.5	7.5	7.5
4	SLES	10	10	10	10	10
5	Sodium sulphate	05	05	05	05	05
6	Urea	03	03	03	03	03
7	Sorbitol (70%)	10	10	10	10	10
8	Polymer (S1) (80.05 %)	3.0	6.0	9.0	12	15
9	Water	49.5	49.5	49.5	49.5	49.5

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