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Production of Cellulose Acetate from Jute Sticks

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A method for the production of cellulose acetate from the jute sticks, *Corchorus capsularis*: late variety -1 (CVL - 1), Fulguni Tossa (0-9897), and BJC 718, was described. Alphacelluloses were isolated from the jute sticks by a standard method. The method of acetylation of the alphacelluloses was then standardised by using acetic anhydride (310% on the dry weight of the material, i.e. alphacellulose,on the weight of material, o.w.m.), glacial acetic acid (410% o.w.m.) and sulphuric acid under the specified conditions. The yields of cellulose acetate were found to be: CVL-1: 55.80%, 0-9897: 45.60% and BJC 718:63.74% on the dry weight basis on the respective jute sticks. The yields were compared with the previous results and found to be greater in the present investigation. The degree of substitution (D.S) for each of the three acetates was found to be around two.

Key words : Jute sticks, Cellulose acetate, Acetylation, Yield.

Introduction

Jute stick is a renewable agro-waste (Sen *et al* 1966), the stick to fibre yield being 2:1. The jute stick, unlike its fibre has no significant commercial value. Researches are, therefore, conducted to convert jute sticks into useful products such as pulp and paper (Sanyal and Day 1982) particle board and charcoal (Banerjee and Bhargava 1980) viscose rayons (Saha *et al* 1973a) and oxalic acids (Raquibdowla *et al* 1969; Mathew *et al* 1984).

Cellulose acetate is usually obtained from cotton linters and wood chips (Doree 1947; Malm 1962). It has many uses such as in production of plastic film, lacquers, photographic films, thermoplastic moulding, transparent sheeting, protective coating solution, cigarette filters, buttons, knobs, pipes, golf tees, camera accessories, magnetic tapes, osmotic cell membranes, combs, telephone, electrical parts etc. But the conventional raw materials used to produce cellulose acetate often fall short of supply, consequently, decreasing the rate of production. Hence availability of the alternative sources of the raw materials is essential. Bangladesh and India, the two major jute producing countries of the world import cellulose acetate to meet the domestic needs. Efforts were therefore, made to convert low-priced jute sticks (costing TK. 0.7/kg in Bangladesh and Rs.0.25/kg in India) to either pulp or cellulose and then into cellulose acetate (Krishnan and Sarkar 1984) which could be an important import substitute to save valuable foreign exchange in Bangladesh and India. The work was limited to the jute sticks of Indian origin. It was observed that jute stick of 40% alphacellulose content can yield 43.5% cellulose acetate on weight basis of stick.

The *Corchorus capsularis*, late variety-1 (CVL-1), Fulguni Tossa (designated by 0-9897) and BJC 718 (previously designated as S718) are three high yielding varieties of jute for fibre of high quality and are being widely cultivated. The 0 - 9897 is a cross (x) of Bangladesh olitorius-5 with Brazilian variety of jute (i.e.0-5xBZ). The BJC 718 is a cross (x) of Dhaka-154 and Capsularis Blue seed (i.e. D-154 x CBS). In our earlier communication (Amin and Shahjahan 1994), we had reported that the sticks of these three varieties of Bangladesh origin had higher cellulose than that of the corresponding Indian jute sticks (Table 1) Hence, it was presumed that the higher alphacellulose content of jute sticks of these varieties may have a certain relation with the yield production of cellulose acetate. In view of this, the present work was initiated to convert the jute sticks of CVL-1, 0 - 9897 and BJC 718 into cellulose acetate.

Materials and Methods

Samples of jute sticks. Jute sticks of cultivars, CVL-,1 0 - 9897 and BJC 718 were collected from the experimental field of Manikgonj sub-station of the Bangladesh Jute Research Institute (Bangladesh).

Separation of alphacellulose from stick. Jute sticks were cut into pieces and ground to powder. The cellulose of each sample was isolated by using the TAPPI method (Timell 1957). The results are summarised in Table 1.

Preparation of Cellulose Acetate by Using the Alphacellulose.

Acetylation of alphacellulose. Sample of alphacellulose (2g) was taken in a conical flask (250 ml) and kept in an ice

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bath (10°C). A mixture of acetylating ingredients consisting of acetic anhydride (8.2 ml., 310% on the weight of material, o.w.m. i.e. alpha cellulose), glacial acetic acid (8.24 ml, 410% o.w.m.) and concentrated sulphuric acid (0.17 ml, 8.5% o.w.m) was prepared and cooled (at 10°C), poured drop-wise in the conical flask with constant stirring and then kept overnight for cooling with occasional stirring. The opaque pasty mass thus obtained was warmed raising the temperature slowly to 50-55°C under reflux and kept for 24h at this temperature until the mass became more or less transparent, viscous and light brown in colour. A test portion of this viscous mixture was precipitated with cold water, filtered, washed until neutral and dried. It was insoluble in acetone, but completely soluble in chloroform or a mixture of chloroform and methanol (9:1 $v\,v^{-1}$), which indicated that the acetylation was carried out as per requirement.

Ripening of the acetate by partial hydrolysis. After the acetylation had been carried out as described above, a solution of acetic acid prepared by mixing glacial acetic acid (1.2 ml., 60% o.w.m) and water (1.2 ml, 60%,o.w.m) was added to the viscous mixture with vigorous stirring to avoid precipitation. After a short while, the viscous mixture was again warmed under reflux at 40-45°C (8-9h) taking care that the colour of the viscous mixture did not change i.e. the viscous mixture remained transparent and light brown (A test portion of the mixture, after precipitation with cold water, filtering, washing to neutrality, drying at 35-40°C should be entirely soluble in acetone). After having a positive test the rest of the viscous mixture was filtered through a sintered funnel, if necessary and the filtrate was then poured down into excess cold water. The precipitate of the acetate was filtered, washed to neutrality and dreid (35-40°C). The white powder of cellulose acetate (yield;112.75-134.11% on the weight of cellulose, Table 1) was soluble both in acetone and in a mixture of chloroform and methanol, but insoluble in water, ethyl alcohol, petroleum ether and benzene.

Determination of acetyl contents. The acetyl contents of the acetate derivatives prepared from the respective cellulose of the jute sticks of CVL-1, 0 - 9897, and BJC 718 were determined by standard method (Amin 1985) The results re summerised in Table 1.

Characterisation of the cellulose acetate. The cellulose acetate samples were characterised for an important parameter i.e. the degree of substitution (DS). For this purpose, the DS values of the acetate of CVL-1,0 - 9897 and BJC 718 were evaluated by the modified Eberstadt method (Saha et al 1973 b) by using the following formula.

$$DS = \frac{3.86 \times \%\ \text{acetyl}}{102.4 - \%\ \text{acetyle}}$$

The DS calculated by the above procedure are summarised in Table 1.

Results and Discussion

The commercial cellulose acetate supplies have D S (degree of substitution - average number of acetyl groups per anhydro -D- glucose unit) values in the range of 2.3-2.7 (corresponding acetyl content of 38-42% (Krishnan and Sarkar 1984). Based on the information, the products of the present investigation were assessed in terms of the yield of cellulose acetate, the acetyl content, and the degree of substitution (D.S) of the respective cellulose acetate acetyl content, and the degree of substitution (D.S.) of the respective cellulose acetate derivatives of the jute sticks of CVL-1, 0 - 9897 and BJC 718. Experiments were repeated thrice for each sample. The mean values of the three samples are presented in Table 1. However, there remains the scope of extended statistical analysis in future studies. The results are discussed categorically.

Yield of cellulose acetate. The yields of cellulose acetate samples were estimated on the dry weight basis for both

Table 1

Yield of cellulose acetate of four different cultivars along with respective alpha cellulose, acetyl content and degree of substitution (D S).

Cultivars of the jute stick	Alpha Cellulose %	Cellulose acetate (%)		Acetyl content (%)	Degree of substitution
		Based on alpha cellulose	Based on jute sticks		
Jute sticks of Indian origin	40.00	Note done	43.50	40.25	2.50
CVL-1	45.10	123.70	55.80	33.12	1.8453
0-9897	45.40	112.80	45.60	32.94	1.8305
BJC 718	47.53	134.11	63.74	33.70	1.8935

alphacellulose and jute sticks. The yield ranged from 45.60 - 63.74% on the weight of jute sticks and 112.80-134.11% on the weight of alphacellulose. The BJC 718 variety was found to give the highest yield while the 0-9897 gave the lowest yield. The results were also compared with the percent cellulose acetate obtained on the weight of the jute stick (Table 1) as in the previous investigation (Krishnan and Sarkar 1984). The present investigation indicated better yield for all the three jute sticks. The process therefore appears to be significant.

Acetyl content and degree of substitution. It appears from Table 1 that the acetyl content is lower than that of the earlier work on jute sticks of Indian origin (Krishnan and Sarkar 1984). This can be explained from the values of D.S. From Table 1 it appears that the values of D.S. for the present investigaion range from 1.8305 to 1.8935 (≈ 2.0) while that in earlier work (Krishnan and Sarkar 1984) the value x was 2.5 for acetate derivative. The lower D.S. value possibly led to decrease the acetyl content of the acetate derivatives in the present investigation.

Conclusion

From the foregoing it is apparent that the utility of the jute sticks can be increased if these are subjected to acetylation for the productionof cellulose acetate . The farmers may get double return from the jute cultivation through such efforts i.e. one from the fibre and the other from the jute stick. This may thus encourage the farmers in jute cultivation and also aid to boost up the overall jute based economy.

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