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A COMPARATIVE STUDY OF ACID HYDROLYSIS OF CELLULOSIC WASTE (WASTE OF HOSIERY INDUSTRY) FOR MANUFACTURING MICROCRYSTALLINE CELLULOSE

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ABSTARCT

Waste management is one of the biggest problems faced by the human beings in industry. Hosiery industries are having the abundant waste of the cotton rags and fabric cuttings during the manufacturing the finished products. Near about 8-9% of waste is generated from these industries. As cotton is having highest percentage of cellulose (87-96%), it can be used for manufacturing microcrystalline cellulose (MCC). Microcrystalline cellulose is used in various industries like pharmaceutical, food, cosmetics etc. It can be derived by number of process like reactive extrusion, enzyme mediated, steam explosion and acid hydrolysis. Out of these processes, the acid hydrolysis is used in present study, because it requires shorter reaction time than others. It can be made continuous and it requires less amount of acid and produces smaller particles of microcrystalline cellulose. For this study, three acids have been used, hydrochloric acid (HCl), sulphuric acid (H_2SO_4), and nitric acid (HNO_3) for the production of MCC. To find the best concentration of acid for getting the higher yield in the process, the different concentrations were tried. 2.1N Hydrochloric acid was found to give the highest yield of 93%, 40% (by weight) concentration of sulphuric acid gives the 90% yield and 65% (by weight) concentration of nitric acid gives the 82% yield. The MCC produced from hydrochloric acid is the fine powder and requires no further grinding where as the MCC produced from the other two acids is in the form of white pellets, which requires further mechanical grinding resulting in the increase in manufacturing cost. The FTIR spectrum shows the similarity with the commercial microcrystalline cellulose and is characteristic of cellulose type I.

Key words: Cellulosic waste, Hosiery industry, Microcrystalline cellulose, Hydrochloric acid, Sulphuric acid, Nitric acid.

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INTRODUCTION

Microcrystalline cellulose (MCC) is purified, partially depolymerized cellulose prepared by treating alpha-cellulose, obtained as a pulp from fibrous plant material, with mineral acids. The degree of polymerization is typically less than 350. It is a naturally occurring substance that is proven stable, safe and physiologically inert. Microcrystalline cellulose has revolutionized tableting because of its unique compressibility and carrying capacity. It exhibits excellent properties as an excipient for solid dosage forms. It compacts well under minimum compression pressures, has high binding capability and creates tablets that are extremely hard, stable, yet disintegrate rapidly. Other advantages include low friability, inherent lubricity, and the highest dilution potential of all binders. These properties make microcrystalline cellulose particularly valuable as a filler and binder for formulations prepared by direct compression, although it is also used in wet or dry granulation. As the name implies, microcrystalline cellulose is cellulose derived from high quality wood pulp, while cellulose is the most abundant organic material. Microcrystalline cellulose can only be derived from a special grade of alpha-cellulose.

MCC is produced using different sources like soybean husk, water hyacinth, coconut shell, sugarcane bagasse and jute.

In this study, MCC has been produced from cellulosic waste such as cotton rags from hosiery industries. Three acids such as hydrochloric acid (HCl), sulphuric acid (H_2SO_4), and nitric acid (HNO_3) have been used for the production of MCC. To find the best concentration of acid for the higher yield of the process, different concentrations of acids were tried. The FTIR spectrum shows the similarity with the commercial microcrystalline cellulose and is characteristic of cellulose type I.

EXPERIMENTAL

Reagents and chemicals

The cotton waste was obtained from Rubi Mills Private Limited, Mumbai having 100 % cotton. Hydrochloric acid, sulphuric acid and nitric acid were of analytical reagent grade supplied by S.D. Fine Chemicals (India), Mumbai. These chemicals were used as received without further purification.

MCC preparation

(i) Using hydrochloric acid

For the manufacture of MCC from HCl and cotton waste, HCl and cotton waste samples were taken in ratio 20 : 1 i.e. 20 mL of HCl acid for 1 g of cotton waste. Acid was heated up to 90^0C and the cotton waste sample was added with continuous stirring in the stirred acid and it was continued up to complete dissolution of waste sample at constant temperature of 90^0C. The time required is nearly 40-45 minutes. The dissolved sample was separated from the acid and the acid was recovered. Then sample was water washed up to neutral pH of the water. The water washed sample was separated from the water and sample was sent for acetone washing. After that, the sample was dried in the pan dryer at 50^0C for one hour and the dried sample was weighted.

For finding the best concentration of acid, different normalities of acid samples were taken (2N, 2.1N, 2.2N, 2.3N, and 2.4N) and the same process was repeated for other four samples.

The typical data obtained on lab scale in preparation of MCC using HCl are given in Table 1.

Table 1

Normality of acid (N)	Volume of acid taken (mL)	Weight of waste sample (g)	Volume of water for washing (mL)	Volume of acetone for washing (mL)	Weight of MCC obtained (g)	% Yield
2.0	200	10	5000	20	8.5	85
2.1	200	10	5000	20	9.3	93
2.2	200	10	5000	20	8.2	82
2.3	200	10	5000	20	8.8	88
2.4	200	10	5000	20	9.0	90

(ii) MCC using sulphuric acid

The production of MCC from sulphuric acid and cotton waste was carried out in the same way as MCC produced from HCl and cotton waste i.e. 20 : 1 ratio means 20 mL of sulphuric acid for 1 g of sample. Different concentrations of acid were used for finding the best concentration of acid required. The concentrations used were 40%, 42%, 44%, 46%, and 48% by weight.

The typical data obtained on lab scale in preparation of MCC using H_2SO_4 are summarized in Table 2.

Table 2

Con. of acid (Weight %)	Volume of acid taken (mL)	Weight of waste sample (g)	Volume of water for washing (mL)	Volume of acetone for washing (mL)	Weight of MCC obtained (g)	% Yield
40	200	10	5000	20	9.00	90
42	200	10	5000	20	8.82	88.2
44	200	10	5000	20	7.62	76.2
46	200	10	5000	20	8.05	80.5
48	200	10	5000	20	7.50	75

(iii) Using nitric acid

The production of MCC from nitric acid and cotton waste was carried out with the same procedure as MCC produced from HCl and cotton waste i.e. 20 : 1 ratio means 20 mL of nitric acid for 1 g of sample. Different concentrations of acid were used for finding the best concentration of acid required. The concentrations used are 50%, 55%, 60%, 65% and 70% by weight

The typical data obtained on lab scale in preparation of MCC using HNO_3 are tabulated in Table 3.

Table 3

Con. of acid (Weight %)	Volume of acid taken (mL)	Weight of waste sample (g)	Volume of water for washing (mL)	Volume of acetone for washing (mL)	Weight of MCC obtained (g)	% Yield
50	200	10	5000	20	8.07	80.7
55	200	10	5000	20	8.00	80
60	200	10	5000	20	7.76	77.6
65	200	10	5000	20	8.20	82
70	200	10	5000	20	7.87	78.7

MCC Analysis

The FTIR analysis was done at Laben Laboratories, Akola using JASCO FT/IR, Fourier Transform Infrared Spectrometer, Model.

RESULTS AND DISCUSSION

The MCC obtained from hydrolysis with hydrochloric acid was white coloured and in powder form; hence, there is no need of further grinding by mechanical means. It can be seen from Table 1 that the highest yield of 93% is obtained from the 2.1 N concentration of hydrochloric acid. So, it is concluded that the optimum concentration of HCl for higher yield is 2.1N HCl.

The MCC obtained from hydrolysis with sulphuric acid was also white in colour but in the form of pellets and not in powder form; hence, further grinding by mechanical means is required, which results in the increase of production cost. It can be seen from Table 2 that the highest yield of 90% is obtained from the 40% (by weight) concentration of sulphuric acid. So, it is concluded that the optimum concentration of H_2SO_4 for higher yield is 40% (by weight). The time required for this hydrolysis is less as compared to hydrolysis with hydrochloric acid and there is no need to supply heat to this process with sulphuric acid as it is exothermic in nature at atmospheric temperature and the temperature of reactor is raised up to 132^0C. Due to high temperature, the cotton waste dissolves faster in this case than the hydrochloric acid.

The MCC obtained from hydrolysis with nitric acid was also white coloured but in the form of pellets and not in powder form; hence, there is a need of further grinding by mechanical means. It results in the increase of production cost. It can be seen from Table 3 that the highest yield of 82% is obtained from the 65% (by weight) concentration of nitric acid. Therefore, it is concluded that the optimum concentration for higher yield is 65% (by weight). The time required for this hydrolysis was more than the other two processes. This is because that the care should be taken during the rise of temperature of nitric acid as it starts to decompose above 85^0C resulting in the formation of nitrogen dioxide (NO_2) fumes, which is toxic to human beings.

The FTIR spectra of MCC obtained with different acid sample are shown in Fig. 1 to 6. All the spectra were compared with the standard microcrystalline cellulose samples and show good results. Only the sample of nitric acid show slight different nature of the spectrum than that of standard spectrum. This may be due to the variation in reactor temperature during the process as nitric acid is temperature sensitive, which results in nitrogen dioxide (NO_2) fumes.

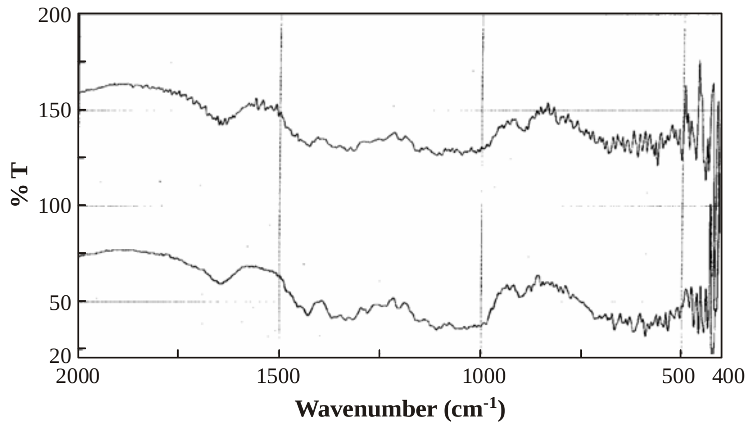

Fig. 1: FTIR spectra for MCC with 2.4 N hydrochloric acid

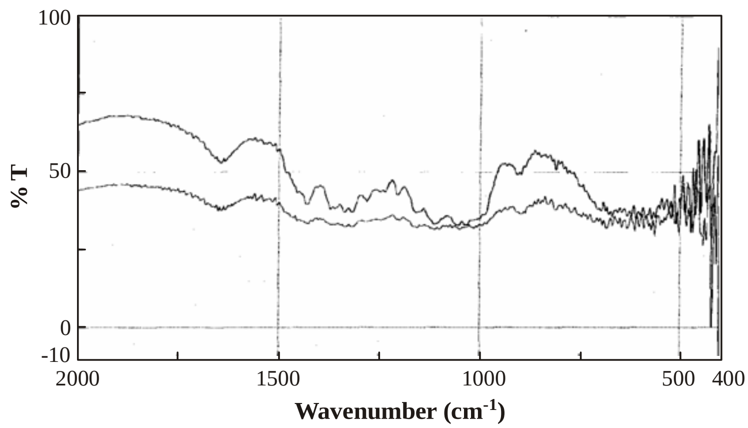

Fig. 2: FTIR spectra for MCC with 2.1 N hydrochloric acid

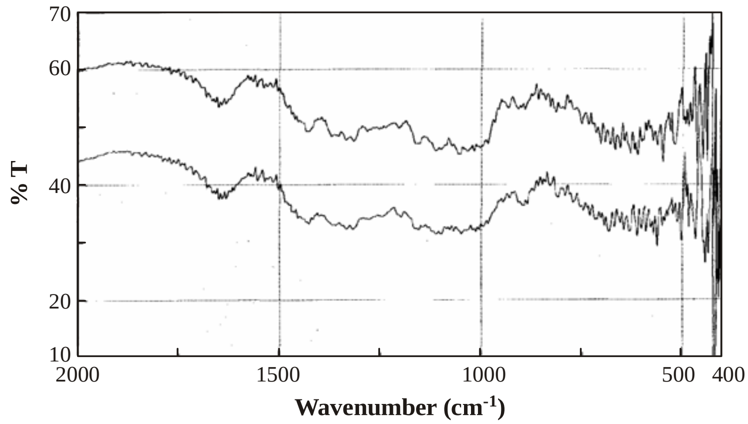

Fig. 3: FTIR spectra for MCC with 48% (by weight) sulphuric acid

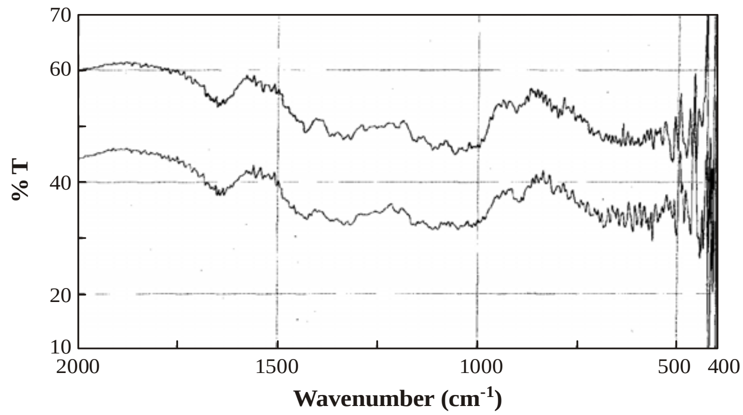

Fig. 4: FTIR spectra for MCC with 42% (by weight) sulphuric acid

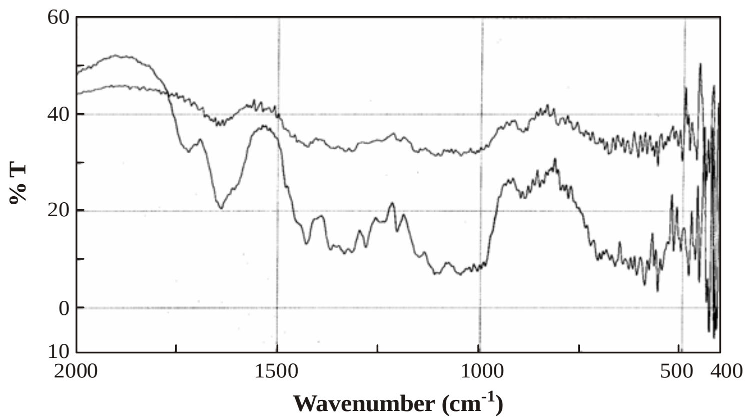

Fig. 5: FTIR spectra for MCC with 70% (by weight) nitric acid

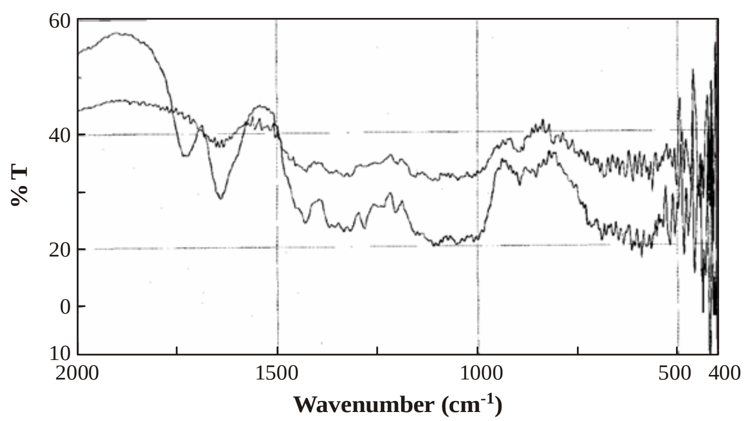

Fig. 6: FTIR spectra for MCC with 55% (by weight) nitric acid

CONCLUSION

MCC prepared in this study from cellulosic waste (waste of Hosiery Industry) has properties similar to that of commercial MCC. The optimum concentrations of hydrochloric acid, sulphuric acid and nitric acid was found to be 2.1N, 40% (by wt.) and 65% (by wt.), respectively, while the maximum yields obtained were 93%, 90% and 82% for hydrochloric acid, sulphuric acid and nitric acid, respectively. The nature of MCC found in powder form for hydrochloric acid while, for sulphuric acid and nitric acid, it was obtained in the form of pellets. The FTIR spectra show similar properties to commercial MCC for hydrochloric acid and sulphuric acid while for nitric acid, it shows some differences. Residues from different plants such as sugar cane bagasse, cereal straws, corncobs, cereal husks, agricultural residues etc. are interesting alternatives as cellulose source for several applications. Such materials are renewable and widely available in many parts of the world. Normally, these are generally burnt or disposed for ambient degradation.

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