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Modification of Cassava Residue into Carbomethylcellulose

[1]P.U. Ofora, [1]R.U. Arinze, [2]J.N. Nwokoye, [1]C.T. Onyema, [1]I.J. Ekwueme and [1]C. Okpalaunegbu

[1]Department of Pure and Industrial Chemistry, P.M.B. 5025, Awka, Nigeria
[2]Department of Chemistry, Federal College of Education, Umunze, Nigeria

Abstract: The residue from cassava starch extraction was chemically modified into carboxymethylcellulose (CMC). The produced CMC was of good quality when compared with the commercial brand in terms of solubility, pH and viscosity respectively. Fourier Transform Infrared Spectroscopy on the produced CMC and commercial grade showed the presence of hydroxyl, ethers, esters and alkyl functional groups and absence of cyanogenic functional group; which confirmed that the modified cassava starch residue is safe for human and animal consumption hence suitable for pharmaceutical and baking applications.

Key words: Cassava residue · Carboxymethylcellulose · pH · Solubility and viscosity

INTRODUCTION

Cassava, *Manihotesclenta Crantz* (syn. *Manihotutilissina Pohl)*, is a dicotledonous perennial plant belonging to the botanical family *Euphorbiaceae*. It is a starchy root crop that is grown almost entirely in the hot lowland tropics. Cassava is a very versatile commodity with numerous uses and by- products [1]. The cassava roots contain hydrocyanic acid, the quantity being a characteristic of the bitter and sweet varieties. Thus, the three varieties of cassava under study are; white petoils,(A), red petoils, (B) and improved varieties, (C). The varieties depend on the presence of cyanogenic glucosides which give the characteristic tastes in the fresh cassava roots [2]. Cassava is processed leaving huge amount of residue from cassava starch extraction which is usually discarded. The cassava fibrous residue (CFR), which constitutes about 15-20% by weight of the processed cassava roots is retained on sieves during the rasping process. It contains about 55-65% starch (on dry weight basis) [3,4]; hence, the CFR constitutes an industrial waste which is a loss to the producer and a nuisance to the environment during disposal [5].

But this can be modified into chemical compounds for industrial applications. Thus, an attempt has been made to utilize this waste, obtained from three cassava varieties, in the production of carboxymethylcellulose (CMC) and compare the qualities of the produced CMC with the commercial CMC.

Carboxymethylcellulose is a water- soluble gum and was formerly referred to as cellulose glycolate. The commercial and industrial applications of CMC continue to increase, due to new developments in foods, pharmaceuticals, cosmetics, textile, paper, adhesives, ceramics and oil well-drilling fluids [6].

MATERIALS AND METHODS

Materials: The cassava roots of the three varieties,whitepetoils, A, red petoils, B and improved variety, C, were collected from the farm in Awkuzu, Oyi Local Government Area,Anambra State, Nigeria. The freshly harvested roots were washed, peeled, washed again and then grated and milled with an electric grinding machine designed specifically for crushingof cassava roots. The resulting pulp was mixed with a large quantity of water and sieved with a sieving cloth. The cassava residue was retained on sieves during the rasping process. This was done for the other varieties. The residue was dried, ground to a mesh size of 150µm and poured into an air- dry container. It was kept ready foruse in the modification and analysis

The following Analytical Grade Reagents were used for the preparation of the CMC:

Monochloroacetic Acid, Sodium Hydroxide, Isopropanol, Deionized Water

Methods: Cassava residue was weighed and poured into a beaker. Water and isopropanol mixed in the ratio of 50:50 at room temperature were poured into reaction flask. This was set-up in a water bath and the temperature controlled at 45°C. The cassava residue sample was poured into the

Corresponding Author: P.U. Ofora, Department of Pure and Industrial Chemistry, P.M.B. 5025, Awka, Nigeria.

reaction flask at a temperature of 45°C, stirred and was allowed to heat for 20 minutes. Then, the prepared solution of sodium hydroxide was added to the reaction mixture while stirring. After 15 minutes, monochloroacetic acid was added and this started the etherification reaction. The reaction mixture was heated for 5 hours at the controlled temperature of 45°C with continuous stirring. The mixture was filtered, the residue washed thoroughly with methanol, the pH was checked to be neutral and dried in a solar dryer. The sample was ready for analysis.

Quality Tests on the Produced Carboxymethylcellulose: Each sample of the produced carboxymethylcellulose was tested for the following qualities and compared with the commercial brand, used as a standard.

pH Determination: The produced carboxymethylcellulose was dissolved in water at room temperature and the pH was measured using a Jenway pH meter, model 3510.This was done three times for each of the samples and the average was recorded.

Solubility Test: Each of the test samples was dissolved in 20ml of water at roomtemperature. The solubility was determined after one hour, thirty minutes (1 h 30mins) and the values obtained were recorded.

Viscosity Measurement: The test sample was dissolved in water at room temperature and the viscosity was determined. The values were calculated using the formular;

Relative Viscosity = t/to

Where t is the efflux time for the solution
To is the efflux time for the solvent.

Spectral Analysis on the Produced CMC and Commercial Brand: The Infrared spectra of the samples and commercial brand were determined using Fourier Transform Infra Red spectrophotometer to ascertain the main functional groups present in the samples. The samples prepared in pellets form mixed with potassium bromide crystals, placed in the KBr cells and inserted into the sample compartment of the spectrophotometer. The spectra were printed out which showed the specific absorption patterns in terms of percentage transmittance against wave numbers. Interpretation of the finger prints showed the functional groups present in the produced and standard samples [7].

RESULTS AND DISCUSSION

The results of the quality control tests and spectra analysis carried out on the test and standard samples are stated below.

The values are the means of the three determinations.

It is evident from Table 1 above, that the quality tests carried out on the three varieties of cassava residue modified into carboxymethylcellulose showed remarkable qualities when compared with commercial brand used as the standard. The produced CMC are neutral salts as was seen from the values of pH measurement. Thus, they are safe to be used.

The solubility and viscosity tests showed that the test samples are good water soluble gum, as well known of carboxymethylcellulose. They dissolved completely in water forming thick viscous gum.

It was observed from the Table 1 above, that cassava residue of red petoils sampled as B, exhibited the best qualities and yield, among the three varieties. While sample C showed better results than sample A. The yield after production was also remarkable. Hence, the cassava residue can be used in the production of carboxymethylcellulose, which has vast applications. This will reduce environmental pollution from the discarding of the residue, increase economic yield and improve the cultivation of cassava.

The functional groups present in the standard CMC were seen to be alkyl, hydroxyl, ether and esters groups, as shown in Table 2. These linkages confirm the different functional groups present in the structure of carboxymethylcellulose.

Table 3, shows the functional groups present in the produced cassava residue A as the alkyl, hydroxyl, carbonyl and ether linkages. It is evident, that similar functional groups were also present in the standard CMC, except in the presence of carbonyl groups (alkanals and alkanones).

It is clear from Table 4, that all the functional groups present in the produced cassava residue B CMC were also present in the standard CMC, except in the presence of carbonyl groups (alkanals and alkanones).

A Similar trend in the spectral analysis of cassava residue C with that of cassava residue B is also observed as shown in Table 5, where all the functional groups present in the produced cassava residue C CMC, were also present in the standard CMC, except in the presence of carbonyl groups (alkanals and alkanones). The presence of carbonyl groups in the produced cassava residue CMC samples could be attributed to the organic nature of cassava tubers while the standard CMC is a chemical product.

Table 1: Physical Characteristics of Produced and Standard CMC

Test Samples	CMC Residue A	CMC Residue B	CMC Residue C	Standard CMC
Mass of Samples (g)	2	2	2	2
Volume of Water (ml)	20	20	20	20
pH Values	6.75	6.92	6.80	7.41
Solubility (gcm^3)	0.07	0.09	0.08	0.10
Viscosity	0.45	0.62	0.48	0.73
% Yield	68	75	70	
Remarks	Good	Better	Good	

Table 2: FTIR Spectral Analysis of Standard CMC

Wave Number (cm^{-1})	Functional Groups	Inference
3697	O-H stretch free	Alkanols,ROH
2932	C-H stretch	Alkyl groups CH_3,CH_2,CH
1240	COO- Stretch	Alkanoates RCOOR’
1100	C-O stretch	Ethers, R-O-R’

Table 3: FTIR Spectral Analysis of Produced CMC A

Wave Number (cm^{-1})	Functional Groups	Inference
3447	O-H stretch free	Alkanols, ROH and Phenols, AROH
3015-2972	C-H stretch	Alkyl groups CH_3,CH_2,CH
2515	Strongly –OH- bonded	Alkanols, ROH
1990-1632	C=O Stretch	Carbonyls, RCHO, RCOR’
1260-1125	C-O stretch	Ethers, R-O-R’

Table 4: FTIR Spectral Analysis of Produced CMC B

Wave Number (cm^{-1})	Functional Groups	Inference
3748	O-H stretch	Alkanols, ROH
3449	O-H stretch free	Alkanols,ROH,PhenolsAROH
2960-2871	C-H stretch	Alkyl groups CH_3,CH_2,CH
2517	Strongly –OH- bonded	Alkanols, ROH
1638	C=O Stretch	Carbonyls, RCHO, RCOR’
1276-1258	COO- stretch	Alkanoates, RCOOR’
1216-1022	C-O stretch	Ethers, R-O-R’

Table 5: FTIR Spectral Analysis of Produced CMC C

Wave Number (cm^{-1})	Functional Groups	Inference
3743	O-H stretch free	Alkanols, ROH
3460	O-H stretch	Alkanols, ROH and Phenols ArOH
2962	C-H stretch	Alkyl groups CH_3,CH_2,CH
1987	C=O Stretch	Carbonyls, RCHO, RCOR’
1265	COO-	Alkanoates, RCOOR’
1136- 1066	C-O stretch	Ethers, R-O-R’

The Fourier Transform Infra-red spectroscopy analysis showed the frequencies and corresponding functional groups present in the samples in the region of very prominent and sharp peaks. The spectral data of the produced CMC from cassava residue showed the presence of some functional groups in the region of sharp and prominent peaks as; hydroxyl groups,-OH, alkyl groups,-CH_3, carbonyl groups, RCHO and RC=O, ethers,-RCOR’ and alkanoates,-RCOOR’. While the commercial grade showed the presence of hydroxyl group-OH, alkyl group-CH3, acetate RCOOR and ethers, ROH, in the region of sharp, strong and prominent peaks. The produced CMC test samples showed the presence of the same functional groups with commercial grade.

CONCLUSION

The study on the three, 3, different species of cassava residue namely white petoils,A, red petoils, B and improved variety,C, modified into carboxymethylcellulose showed that species B had tremendous qualities compared to the commercial grade, used as the standard. While species A and C also showed remarkable results. Furthermore, the spectral data of the test samples showed the presence of almost the same functional groups with the standard, CMC. These data confirmed that cassava residue can be modified into carboxymethylcellulose, when related and compared to the results obtained from the standard CMC.

Thus, the modification of cassava residue into carboxymethylcellulose gave results that showed an important economic value of this agricultural waste (that is usually discarded), leading to environmental pollution.

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