

PRODUCING OF SOME DERIVATIVES OF CELLULOSE BY RECYCLING OF PAPER AND AGRICULTURAL WASTES

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ABSTRACT

The world now is looking for renewable, cheap alternatives to be used in industry to save the environment for sustainable life and the money for the factory's owners. This research is dealing with the possibility of synthesizing nitrocellulose (NC) from an easily renewable agriculture waste such as bagasse, is demonstrated in extraction of α -cellulose, by pulping, bleaching, and nitration, were found nitrocellulose with nitrogen content ranged from (less than 7.0 to 11.4 %), and relative viscosity 1% from (1.09 to 4.44). The NC structure and fibers were characterized by analytical techniques such IR spectroscopy and proton NMR spectroscopy. It was found that the bagasse gives very good results when used to give nitrocellulose.

Keywords: alpha cellulose – nitrocellulose- bagasse.

INTRODUCTION

Continuation to our scholar researcher work the author enhance the economic value of agriculture wastes to minimize the pollution environmental impact of the open fire agriculture waste. (El Kady *et al.*,

2014; El Hoshady *et al.*, 2015; El Kady *et al.*, 2016 and Saleh *et al.*, 2019).

The volume of the agricultural wastes (leaves, tree cuts, crop residues, husks or roots) is estimated by about 32-35 million tons per year (Abdel-Ala *et al.*, 2003; Abou Hussein and Sawan 2010 and Bayoumi *et al.*, 2014).

The misused of these agricultural wastes represents a dangerous environmental damage and a waste of an economic resource.

Agricultural wastes such as wheat straw, rice straw, bagasse, and cotton stalk are rich in lignocellulose and primarily contain cellulose, lignin, hemicellulose, and extractives (Khezami *et al.*, 2005 and Stefanidis *et al.*, 2014). Cellulose forms a skeleton that is surrounded by hemicellulose and lignin functioning as matrix and encrusting materials, respectively (Ingram and Doran 1995).

The environmental impact due to bagasse pulp production is more controlled Compared to rice straw pulping, due to the different chemical properties of the effluents. Although rice straw is available at a minimal cost, the economic effectiveness is relatively low due to unavailability of proven technology for chemical recovery of silica content from the resulting black liquor. Furthermore, a typical mill for soda or kraft pulp using bagasse in Egypt generates energy in the form of steam and electricity from recovery boilers (EEAA, 2003).

Producing of some derivatives of cellulose by recycling of bagasse were done in our research by soda pulping mechanism.

We tried to close our work on the chosen raw materials to the way of handling the cotton linter in industry, to ease and faster the substitution of cotton linter by our chosen raw materials according to the results obtained.

Nitrocellulose (NC), is an important industrial polymer manufactured by nitrating the hydroxyl groups of cellulose. It has many applications, the nature of which depends on its degree of nitration or nitrogen content. At low nitrogen contents (below 12.2 %) it is used in cosmetics, printing inks, paints and lacquers, whereas at nitrogen contents above 12.2 %, it is used as an energetic ingredient in gun and rocket propellants . Moreover, NC has been used in a wide range of products including plastics, paints and lacquers, and smokeless gun propellants (Gismatulina *et al.*, 2017).

The aim of the work is to produce cellulose and some of its derivatives such as nitocellulose from a renewable, cheap agriculture waaste alternatives such as bagaase.

MATERIALS AND METHODS

Materials: Sodium hydroxide, pellets, 98%, Dae-Jung, reagents chemicals, Kosoq, listed company, Hydrogen peroxide solution 30%, PDH laboratory supplies pools, England, Diethyl ether, Abo Zaabal Company for specialized chemicals. Acetic acid (glacial, AR grade), Carbon group, Cork,

Ireland, Copper II ethylene diamine solution, PDH laboratory supplies, England, Ferric sulphate hydrate extra pure, assay as Fe 20%, LOBA chemie, Acetone pure 99% produced by Abo zaabal company for specialized chemicals, Sulphuric acid 98%, made in United Kingdom, Merck KGaA, Germany.

Preparation of the raw material: The stalks of bagasse were cut into small pieces ranged from 3.0- 5.0 cm, in some experiments the outside green bark was removed, and the stalks were grinded to fine powder.

Pulping process: Used alkaline pulping process which include the delignification of cellulosic fibers by alkaline sodium hydroxide concentration 17.5% (gm volume) in molar ratio 1:5 to 1:10 gm m.volume either by refluxing on water bath or by put in round glass equipped with water condenser refluxed from 4-9.5 hrs. with vigorous shaking from time to time, at the end of time filter the content in centery glass, washed with tab water, then distilled water till neutralization, finally washed by aqueous acetic acid to completely neutralize, then ethyl alcohol dry the content, gravimetrically estimate the yielding percent and stored into polyethylene bags to the next step process and easily removed the hemicellulose to convert the produced cellulose into fibers.

Table (1): preparing and pulping conditions of bagasse

Exp. No.	Preparation conditions	Pulping conditions with NaOH%
1	Cuttet bagasse, bag in a water bath	(Wt. of sample: sod. Hydroxide 17.5% 1:10) for 3.5 hrs. Then renewing the soln. for 2 more hrs.
2	Flatted, cut sample – on an oil bath	(Wt. of sample: sod. Hydroxide 17.5% 1:8) for 4 hrs.
3	Flatted, cut sample - on a water bath	(Wt. of sample: sod. Hydroxide 17.5% 1:8) for 5 hrs.
4	Flatted, cut sample - on a water bath	(Wt. of sample: sod. Hydroxide 17.5% 1:8) for 5 hrs.
5	Debarked, cut sample, on a water bath	(Wt. of sample: sod. Hydroxide 17.5% 1:8) for 5 hrs.
6	Cut, grinding sample, on a water bath	(Wt. of sample: sod. Hydroxide 17.5% 1:8) for 5 hrs.
7	Grinded barked, sample, on a water bath	(Wt. of sample: sod. Hydroxide 17.5% 1: 5) for 5 hrs.
^	the sample was debarked and grinded, on a water bath	(Wt. of sample: sod. Hydroxide 17.5% 1: 10) for 9.5 hrs.

Treatment with H₂SO₄: For some experiments, pulped bagasse was treated with aqueous sulphuric acid 1% refluxing for 10 minutes, 20 minutes, 30 minutes and an hour. Determination of the loss content (weight of the sample, weight of the residue remained after treatment), comparing the α -cellulose content.

This treatment to fibrate and increase the delignification to activate the raw materials towards nitration process.

In exp. no. (8), the pulped sample was divided into 5 groups, 4 of them treated with 1% H₂SO₄. For different times, and the fifth processed normally

without adding H₂SO₄, exp.no. (8-b) gives the best results according to Wt. remains after adding H₂SO₄, as shown below in table no. 2

Table (2): H₂SO₄ treatment conditions on pulped bagasse

Exp. No.	1% H ₂ SO ₄ treatment conditions	Wt. remains after adding H ₂ SO ₄ only %
8-a	10 min. On a water bath,	84.2
8-b	20 min. On a water bath,	86.0
8-c	30 min. On a water bath,	81.4
8-d	1 hr. On a water bath	81.8
8-e	Without adding H ₂ SO ₄	_____

Bleaching: Selected pulp samples were bleached in 2 stages, The first stage proceeded by aqueous sodium hypochlorite (4%) for about 2.5-5 hours then washed with tap water till neutralizing. The second stage proceeded by bleaching the first stage samples with mixture of hydrogen peroxide (2%), and aqueous solution of sodium hydroxide (2%) 1:1, for about 3-5 hours with flipping over from time to time at ambient room temperature, and then washed with tap water till neutralizing.

As we will see in table (3), the best conditions of pulping and bleaching of bagasse according to the highest yield of Wt. remains after bleaching % is exp. no. (4).

Table (3): bleaching conditions of bagasse

Exp. No.	Bleaching 1st. step	Bleaching 2nd. step	Wt. remains after bleaching %	Observ.
1	3 hrs.	5 hrs.	27.18	Unbulky sample has some unchanged parts, has buff color
2	4 hrs.	5 hrs.	29.26	Bulky sample , much whiter than exp.3
3	4 hrs.	5 hrs.	31.31	Bulky sample
4	4 hrs.	5 hrs.	33.9	
5	3 hrs.	3 hrs.	23.38	
6	2.5 hrs.	3 hrs.	21.0	
7	4 hrs.	3 hrs.	23.62	
∧	5 hrs.	5 hrs.		
∧-a			17.05	Little bulky
∧-b			17.75	Little bulky
∧-c			16.96	Little bulky
∧-d			18.38	Powder, little darker than exp.no. 8-e
∧-e			21.47	Powder, much darker than exp.no. 8-d

Tests on bleached raw materials: These tests are carried out to distinguish the α -cellulose content according to 18's factory sheet.

1- α -cellulose content: 10 ml of aqueous solution of sodium hydroxide (17.8%) is added to 2 gm. (wt) of a dried sample (16 hrs in 105°C drying oven) in 25ml beaker, stir fast till the sample absorb the soda solution

then let for 20 min. exactly. Stop reaction by using 50 ml of distilled water , filter in a pre-weighted dried porcelain gouch 1G3(w) ,wash with distilled water till neutralizing , wash with acetic acid 2-3 times, wash with boiled distilled water till neutralizing, then put the gouch in 105°c drying oven for 16 hrs, let cool and reweight the gouch with sample (w1)

$$\alpha\text{- cellulose content (\%)} = [(w1 - w) / wt] \times 100$$

2- Degree of polymerization: Dissolve 0.82 gms of dried sample (16 hrs in 105°c drying oven) in 25ml of copper ethylene diamine in a dark brown sealed bottle ,after using nitrogen gas to replace oxygen in the bottle, let the bottle on shaker till dissolve, then apply solution to Abel hood device , measure the time taken by the solution to pass throw the device ,compare time with the polymerization –time table to explain time in a polymerization degree .

3-Whiteness degree: Take the reading of the color tester device LFM1 (B Raive instruments, DR Lunge. weib standard) after calibration by whitness standard slice with optical standard filter Y (Ry=79.3).

4-Nitration of cellulose: According to Urbanski, (1965): The NC was being prepared and stabilized under synthesis conditions required to obtain NC soluble in alcohol ester. The chosen raw materials cellulose was nitrated with mixed acid containing 17 % H₂O, nitric acid and

sulphoric acid, after the reaction was completed, the residue was filtered in a vacuum filter, stabilized and dried.

Characterization of produced nitrocellulose: According to Bofors,(1960)

1-Nitrogen content : Nitrogen of nitrates is determine in the nitrometer by Lunge-Senderholm's method. By shaking a nitrate solution with excess sulphuric acid in the presence of mercuric, nitrogen is quantitatively disengaged as nitricoxide (NO).The pressure exerted by the gas in a given volume is measured. The weight of NO liberated can then be calculated as following :% of Nitrogen= $.0627.v. (\beta \pm p).273 / w. 760. (273+t)$

Where v= volume of the reading tube in ml.

B= barometric pressure in mm. Hg. Corrected to 0°C.

p= pressure difference between air and gas inside the reading tube, in mm. Hg.(+ in the formula, if the gas shows excess pressure, in relation to the air, otherwise-).

w= weight of sample, t= temperature in °C., $.0627.v.273/760$ is constant =k

So % of Nitrogen= $k. (\beta \pm p) / w. (273+t)$

2-Viscosity : Viscometer by Ostwald-Bohme

-Pour out 1.0 gm of dried nitrocellulose into the mixing cylinder equipped with a cork. Add 120 ml. of a mixture of 93% acetone and 7%

water, place the cylinder in the shaking apparatus till dissolving, and let it settle 2 hours. In another measuring cylinder pour so much of the solution that is immersed on the level with 10 by applying vacuum draw the solution above mark A, record the time by the liquid to drop from mark A to mark B. repeat determination by using distilled water at the same temperature.

Viscosity = efflux time of the solution, in sec. / efflux time of the water, in sec.

RESULTS AND DISCUSSION

The yield of α -cellulose%, Degree of whiteness, and Degree of polymerization are summarized in table (4)

Table (4): Results of tests on bleached bagasse

Exp. No.	α -cellulose%	Degree of whiteness	Degree of polymerization	Observation
1	95.70	71.80	Didn't dissolve	
2	97.19	70.50	Didn't dissolve	
3	90.86	73.10	Didn't dissolve	
4	95.27	70.80	Didn't dissolve	
5	88.40	70.20	Didn't dissolve	
6	86.95	70.90	Didn't dissolve	
7	82.85	73.90	Didn't dissolve	
\wedge -a	91.47		Didn't dissolve	without bleaching
\wedge -b	94.19		Didn't dissolve	without bleaching
\wedge -c	93.01		Didn't dissolve	without bleaching
\wedge -d	93.63		Didn't dissolve	without bleaching
\wedge e-1	95.88		Didn't dissolve	without bleaching
\wedge e-2	80.17		Didn't dissolve	α -cellulose% after bleaching

From table (4), the best results of α -cellulose% is exp. no. (2)

Nitration conditions for bagasse cellulose is (1gm. Of bleached sample:
50 gm. Of mixed nitration acid).

Nitration has done on some experiments of bagasse, not all of them

Table (5): Results of nitration of bleached bagasse

Exp. No.	Nitrogen %	Relative viscosity 1%	Wt.% remaining after Nitration	observation
1	10.99	1.30	73.09	The sample has woody impurities
2	11.40	1.74	The wt. slightly increased	The sample has fibers didn't nitrated
4	11.19	4.44	100	Solution of viscosity has woody impurities
5	10.98	1.75	The wt. slightly increased	The sample has less impurities than exp. 4
7	10.62	1.25	57.11	The sample has yellow color and impurities
8-a	Less than 7	1.09	32.71	
8-b	10.23	1.09	33.0	
8-c	9.85	1.09	29.0	
8-d	9.86	1.09	33.50	
8-e	10.23	1.14	57.86	

From table (5), the best results of nitration according to nitrogen content% is exp. no. (2).

Wt.% remaining after Nitration (slightly increased) is a normal phenomena due to the reaction of nitration.

From the above tables, bagasse gives high results of α -cellulose% and good respond to nitration .

Cellulose and nitrocellulose of exp. no.(2) is proved by using IR spectroscopy, and proton NMR spectroscopy as will show bellow.

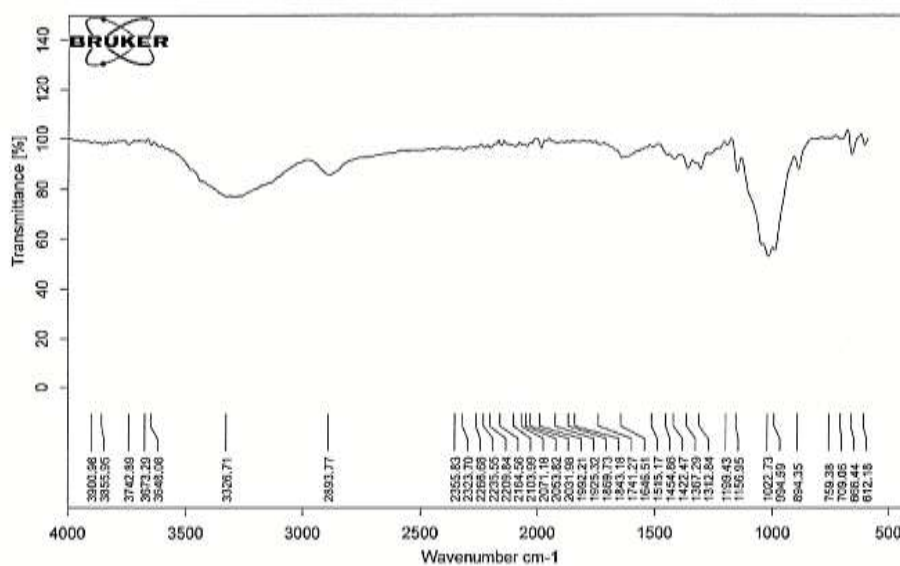


Fig (1): Infra Red (IR) Spectroscopy proves the St. of cellulose from bleached bagasse exp. no.(2)

The structure of cellulose and nitrocellulose, which taken from bagasse, exp. no. 2, have been proven by IR spectroscopy and proton NMR, as follows:

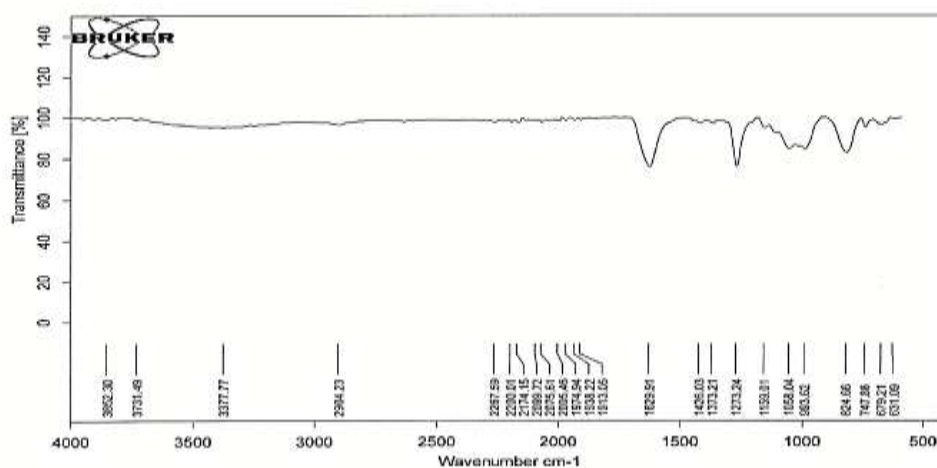
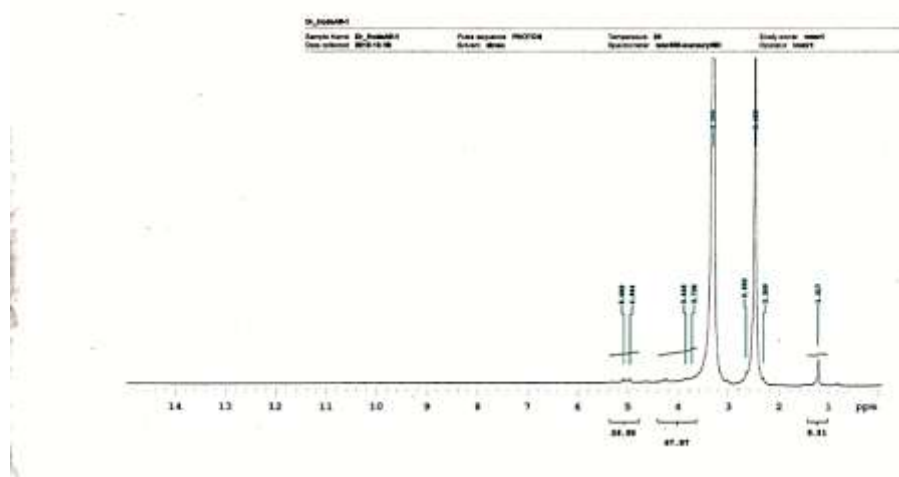


Fig (2): Infra Red (IR) Spectroscopy proves the St. of nitrocellulose from bleached bagasse exp. no.(2)

IR spectra of the bleached bagasse cellulose and nitrocellulose are shown in Figure 1, 2.

A comparison between the IR spectra of the bleached bagasse cellulose and NC shows that stretching peak of hydroxyl groups on the glucose rings near 3500cm⁻¹ was greatly reduced, which signifies that the hydroxyl groups were partially substituted by the nitryl group. After nitration, the spectrum clearly shows the typical signal pattern sought for the NC sample from exp. no. 2. The right half of the spectrum below 1670 cm⁻¹ contains five characteristic peaks: two sharp intense peaks observed at around 1660 and 1280 cm⁻¹ corresponding to the NO₂ asymmetric and symmetric stretching, respectively; a slightly broader, intense peak at around 832 cm⁻¹

1 assigned to the ONO_2 stretching; a less intense peak observed at about 748 cm^{-1} corresponding to the O NO_2 asymmetric bending, while the band at about 684 cm^{-1} is attributed to the O NO_2 symmetric bending. In the vicinity of $1200\text{--}950\text{ cm}^{-1}$ are a few medium-intensity peaks corresponding to different vibrations of the CO group. Overall, the IR spectrum of the bagasse NC exhibits all the peaks representative of nitro groups.



Fig(3): Proton Nuclear Magnetic Resonance (NMR) proves the St. of cellulose from bleached bagasse exp. no.(2)

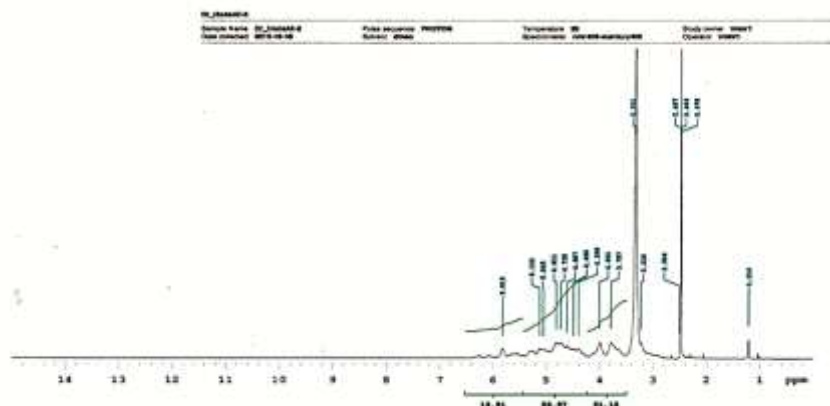


Fig (4): Proton Nuclear Magnetic Resonance (NMR) proves the St. of nitrocellulose from bleached bagasse exp. no.(2)

The ^{13}C NMR spectrum of the bleached bagasse NC exhibits a chemical shift at $\delta = 70.9$ ppm, suggesting that most C-6hydroxyl groups were esterified. Along with that, the C-3and C-2 hydroxyl groups were also substituted by nitro groups, and their peaks appeared at $\delta=83.0$ ppm and $\delta=84.4$ ppm. The peak of the C-6 nitro groups is stronger than those of the C-2 and C-3 nitro groups, indicating that the hydroxyls at C-2 and C-3 were only partially esterified and esterification chiefly occurred on the C-6 hydroxyl groups.

A comparison of the ^{13}C NMR spectrum of the bleached bagasse NC with earlier reported cotton NCs shows the matching of most chemical shifts typical of 6-mono-nitrocellulose, 2,6- dinitrocellulose,3,6-dinitrocellulose,and2,3,6-trinitrocellulose. It is thus evident that the bagasse

NC holds both 2, 6-di-, 3, 6-di- and tri-substituted moieties of the glucopyranose ring of the cellulose macro molecule.

The findings on the synthesis of NC from bagasse are quite promising.

CONCLUSION

This study recommended the uses of cellulosic content of agriculture waste especially bagasse as a raw material for synthesis nitrocellulose instead of cotton linter for a cheap raw material and protection of environmental impact of raw materials.

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إنتاج بعض مشتقات السيلولوز عن طريق المعالجة تدوير الورق والمخلفات الزراعية

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المستخلص

يبحث العالم الان عن بدائل متجددة ورخيصة ليتم استخدامها بالصناعة لتوفر الحماية للبيئة لاستدامة الحياة والمال لاصحاب المصانع. هذا البحث يتناول إمكانية تصنيع النيتروسيلولوز (ن.س) من المخلفات الزراعية المعاد استخدامها بسهولة مثل مصاصة قصب السكر، موضحة من خلال استخلاص الالفا سيلولوز بواسطة التلييب والتبيض والنترنه، والتي تم الحصول فيها على النسبة المئوية لمحتوى النيتروجين للنيتروسيلولوز يتراوح من (أقل من ٧ إلى ١١,٤ ٪) ، واللزوجة النسبية ١ ٪ من (١,٠٩ إلى ٤,٤٤). تم اثبات وجود النيتروسيلولوز بواسطة تقنيات تحليلية مثل

مطيافية الأشعة تحت الحمراء ، مطيافية الرنين المغناطيسي النووي بالبروتون. وجد أن مصاصة
القصب اعطت نتائج جيدة جدا عند استخدامها لانتاج النيتروسيلولوز.
كلمات دالة: ألفا سيلولوز - نيتروسيلولوز - مصاصة القصب.