



# International Journal of Sciences: Basic and Applied Research (IJSBAR)

ISSN 2307-4531  
(Print & Online)

<http://gsrr.org/index.php?journal=JournalOfBasicAndApplied>



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## Investigation of $\alpha$ -Cellulose Local Sources for the Production of Nitrocellulose

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### Abstract

The present study has aimed at investigating some local agricultural wastes as sources for the production of  $\alpha$ -cellulose; these included: sugarcane bagasse, rice straw, durra stalk and groundnut shell. A mixture of toluene and ethanol in the ratio of 2:1 was utilized in solvent extraction, for 6 hr, to remove wax and resins, leaving the fibrous material. This was then delignified by treatment with acidic sodium chlorite ( $\text{NaClO}_2$ ) solution. Alkaline hydrolysis (18% NaOH) was employed to remove hemicelluloses leaving  $\alpha$ -cellulose. The isolated final product was characterized using Fourier Transform Infrared (FTIR) Spectroscopy. The  $\alpha$ -Cellulose content of the four selected plants, (sugarcane-bagasse, rice straw, durra stalk and groundnut shell, were 40.3%, 39.3%, 42.7% and 55.3% respectively. This compares reasonably with the findings of other previous studies, vis. 54.7%, 35.7, 65.8% for bagasse, rice straw, and groundnut shell. From both sets of studies, groundnut shell gave the highest content of  $\alpha$ -cellulose.

**Keywords:** Alkaline hydrolysis; Cellulose; FTIR ; Lignin; Nitrocellulose;  $\alpha$ - cellulose .

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## **1. Introduction**

Cellulose is found in a wide range of species, from higher plants such as wood to green algae; it can be derived from a variety of sources such as wood, seed fiber, grass, algae and even bacteria [1]. However, the  $\alpha$ -cellulose content which is needed for certain applications (e.g. nitrocellulose production) [2], varies widely in these sources. Cellulose is a composite material, and includes (besides  $\alpha$ -cellulose) hemicelluloses, lignin, pectin, wax and a variety of resins [3]. The earliest attempts to isolate  $\alpha$ -cellulose were conducted by the English chemist, they removed undesirable materials by dissolving them in a concentrated sodium hydroxide solution. They specified undissolved residue as  $\alpha$ -cellulose and the soluble materials as  $\beta$ -cellulose and  $\gamma$ -cellulose [4]. Recently the authors in [5] determined the amount of cellulose and four major sugars in cotton fiber. It is also well known that treatment of the lignocelluloses materials with chlorite can remove most of lignin and the following isolation can be performed at room temperature [6].

The authors in [7,8] found that the separation of the cellulose in pulp into  $\alpha$ -,  $\beta$ -,  $\gamma$ -cellulose fractions is an empirical procedure which has been widely used to evaluate pulps for various purposes and obtained two distinct types of hemicelluloses by extraction of wood cellulose with 18.5% NaOH.

Urbanski [9], has shown in 1965, that pure cotton linter gave the highest  $\alpha$ -cellulose content (96%) of all the materials investigated. However, cotton linters would be uneconomical for most of the  $\alpha$ -cellulose applications.

The present study aims at investigating low-cost local cellulose sources that can provide reasonably high  $\alpha$ -cellulose content. The materials chosen (namely, sugarcane bagasse, rice straw, durra stalk and groundnut shell), which may be labeled as "agricultural wastes", are probably the cheapest sources for cellulose.

## **2. Materials and Methods**

Samples of each of the materials chosen (sugarcane bagasse, rice straw, durra stalk and groundnut shell) cut into small pieces, cleaned with distilled water to remove dust and dirt adhering to them, and then dried in an oven at 105°C. The cut, dried samples were ground in an electrical mill to fine powder to increase the surface area.

### **2.1. Solvent Extraction (Dewaxing)**

Each powdered sample was subjected to extraction with toluene-ethanol mixture (2:1) in soxhlet extractor at boiling temperature for 6 hours, so as to remove the extractable materials such as resins waxes, fats and oils. The extract residue (cellulose) was dried at 60 °C for 16 hours and weighed afterwards.

### **2.2. Delignification**

The solid, remaining after extraction with toluene-ethanol mixture, was transferred to a 250-ml flat-bottom flask, 120 ml. of 6% Sodium chlorite ( $\text{NaClO}_2$ ) solution and one ml. of glacial acetic acid were added to the sample to adjust pH at (3.9 –4). The flask was covered, placed on a hot plate, and heated for 2hr at 75°C. The reaction mixture was allowed to cool and equilibrate for 24hr, and then the lignin-containing liquid was decanted. The

decanted liquid placed in a 250-ml flask and 150 ml ice water added. This was repeated five times. The precipitate was placed on a filtration setup and washed with distilled water till the filtrate indicated neutral. The filter paper and residue (lignin) were transferred to a crucible and oven-dried overnight at 65 °C. The lignin-free residue after reaction (holocellulose) was washed oven-dried and weighed.

**2.3. Alkaline Hydrolysis ( $\alpha$ -Cellulose Isolation)**

The dried, delignified sample was transferred to a 250-ml conical flask and 60ml of 18% NaOH was added, and the temperature adjusted to 4C° maintained for 24h. After the reaction completion, the Sodium hydroxide solution was decanted. The solid, which is  $\alpha$ -cellulose, was filtered using pre-weighed filter paper in funnel, and subjected to treatment with glacial acetic acid to neutralize the sodium hydroxide.

The acid was removed by repeated washing with distilled water until the wash water indicated neutral. The  $\alpha$ -cellulose was transferred to a crucible and oven-dried at 70°C to constant weight.

**3. Results and Discussion**

**3.1. Results**

**3.1.1. Chemical Analysis**

The weights of the starting materials, and those obtained at each stage of treatment, have all been obtained on dry basis. The cellulose content of each material was determined as the ratio of the dried lignin free sample to weight of samples expressed as percentages according to this equation:

$$\text{Pure cellulose \%} = \frac{W3}{W1} \times 100 \dots \dots \dots (1)$$

Where: W3 is the weight of oven dried lignin free (purecellulose) sample.

W1 is the weight of oven dried raw sample.

For determination of ash content, Empty crucibles were heated in a muffle furnace at 600°C for 30 minutes, then cooled in a desiccators and weighed. Two grams of each dry sample were placed in the crucible, and crucible with contents ignited in a muffle furnace for 2hr for a final temperature of 600°C. Crucible with its contents was cooled in the desecrator, and then weighed. The ash content of a sample was determined as follows:

$$\text{Ash\%} = \frac{W_f}{W_i} \times 100 \dots \dots \dots (2)$$

Where: W<sub>f</sub> = final sample wt.            W<sub>i</sub> = initial sample wt.

The data obtained on chemical compositions are shown on Tables below:

**Table (1):** Chemical Analysis for total Cellulose Content and Undesirables for the Materials Studied

Material	Sample(gm)	Organic phase extract(gm)	Residue(gm)	Lignin (gm)	Total Cellulose(gm)
Bagasse	3.00	0.42	2.58	0.60	<b>1.98</b>
Rice straw	3.00	0.45	2.55	0.62	<b>1.93</b>
Dura stalk	3.00	0.52	2.48	0.85	<b>1.63</b>
Groundnut shell	3.00	0.31	2.69	0.76	<b>1.93</b>

**Table (2):** Chemical Analysis for total Cellulose Content and Undesirables for the Materials Studied

Material	Sample(gm)	Organic phase extract(gm)	Residue(gm)	Lignin(gm)	Total Cellulose gm
Bagasse	3.00	0.4	2.60	0.61	<b>1.99</b>
Rice straw	3.00	0.44	2.56	0.63	<b>1.93</b>
Dura stalk	3.00	0.59	2.41	0.78	<b>1.63</b>
Groundnut shell	3.00	0.34	2.66	0.75	<b>1.91</b>

**Table (3):** Chemical Analysis of Undesirables for the Materials Studied

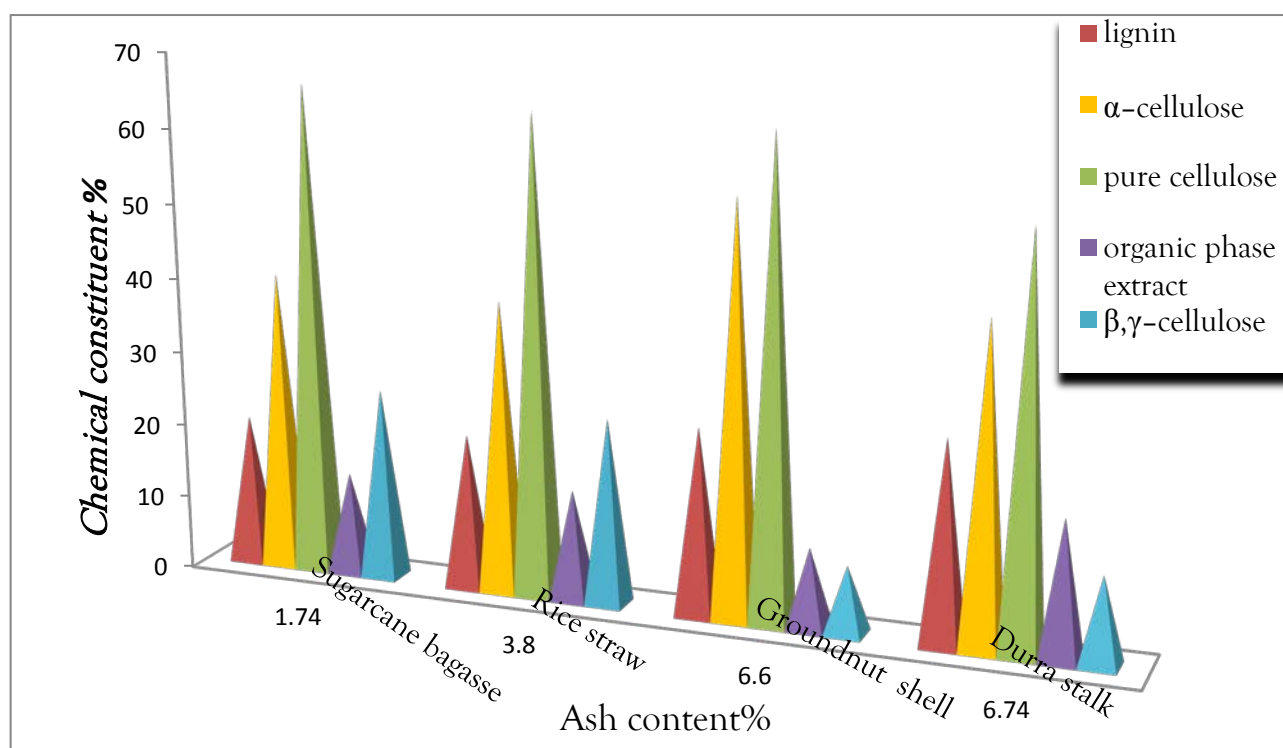
Material	Sample (gm)	Organic phase extract gm(%)	Residue gm(%)	Lignin gm(%)	Ash%
Bagasse	3.00	0.41(13.65)	2.59(86.35)	0.605(20.16)	<b>1.70</b>
Rice straw	3.00	0.445(14.8)	2.56(85.2)	0.625(20.8)	<b>3.6</b>
Dura stalk	3.00	0.555(18.5)	2.445(81.5)	0.815(27.16)	<b>6.76</b>
Groundnut shell	3.00	0.325(10.8)	2.765(89.2)	0.755(25.16)	<b>6.6</b>

Alkaline hydrolysis resulted in the separation of the total cellulose into two parts:  $\alpha$ -cellulose on one hand and ( $\beta$ - +  $\gamma$ -cellulose) on the other. Table (4) shows the percentages of the two parts for each of the materials studied.

**Table (4):**  $\alpha$ -,  $\beta$ -, and  $\gamma$ -Cellulose contents of pure cellulose obtained from the four materials studied.

Material	Sample(gm)	Total Cellulose gm(%)	$\beta$ + $\gamma$ -Cellulose gm(%)	$\alpha$ -Cellulose gm(%)
Bagasse	3.00	1.985(66.16)	0.77 (25.7)	1.21 (40.3)
Rice straw	3.00	1.93 (64.3)	0.75 (25.0)	1.18 (39.3)
Dura stalk	3.00	1.63 (54.3)	0.35 (11.7)	1.28 (42.7)
Groundnut shell	3.00	1.93 (64.0)	0.27 (9.0)	1.66 (55.3)

Table (3)and(4) compares the various contents obtained in total analysis's of the four materials investigated. These are graphically compared to one another and to the ash content in Figure (1) below:



**Fig(1):** Schematic representation of various constituents for each material

### 3.1.2. Spectroscopic analysis:

FTIR spectroscopy of a KBr discs containing finely ground sample was performed in an absorbance mode and the spectrum were recorded and compared that those of the isolated from medical cotton. IR spectra obtained from both raw material and isolated  $\alpha$ - cellulose from each of the four materials are shown in Figures (11 – 22) and listed on Tables 8-11.

**Table (5):** IR patterns for sugarcane bagasse

Peak of raw sample (Cm <sup>-1</sup> )	Transmittance %	Peak of α- cellulose (Cm <sup>-1</sup> )	Transmittance %	Peak of reference (Cm <sup>-1</sup> )	Transmittance %	Functional group
3423.41	68.8	3442.7	51	3368.59	17	OH stretching vibration
2918.10	78.2	2900.74	66	2901.11	31	C-H stretching vibration
1631.67	79.8	1639.38	71.9	1638.36	44.2	H <sub>2</sub> O absorption
1373.22	75.3	1373.22	63	1372.3	28.5	C-H bending
1045.35	56.3	1062.7	47.7	1058.76	14.1	C-O-C bending
902.62	82.7	896.84	69.3	896.52	32.1	C-Ostretch& deformation
655.75	78.8	663.47	64.6	667.84	30.5	O-H bending

**Table (6):** IR patterns for Rice straw

Peak of raw sample (Cm <sup>-1</sup> )	Transmittance %	Peak of α- cellulose (Cm <sup>-1</sup> )	Transmittance %	Peak of reference (Cm <sup>-1</sup> )	Transmittance %	Functional group
3419.56	53.6	3421.48	51.2	3368.59	17	OH stretching vibration
2920.03	67	2914.24	61.6	2901.11	31	C-H stretching vibration
1631.67	66.9	1637.45	64.7	1638.36	44.2	H <sub>2</sub> O absorption
1371.29	65.5	1371.29	61.4	1372.3	28.5	C-H bending
1066.56	49.8	1066.56	53.1	1058.76	14.1	C-O-C bending
900.00	71	895.84	66	896.52	32.1	C-Ostretch& deformation
667.32	67.8	667.32	63	667.84	30.5	O-H bending

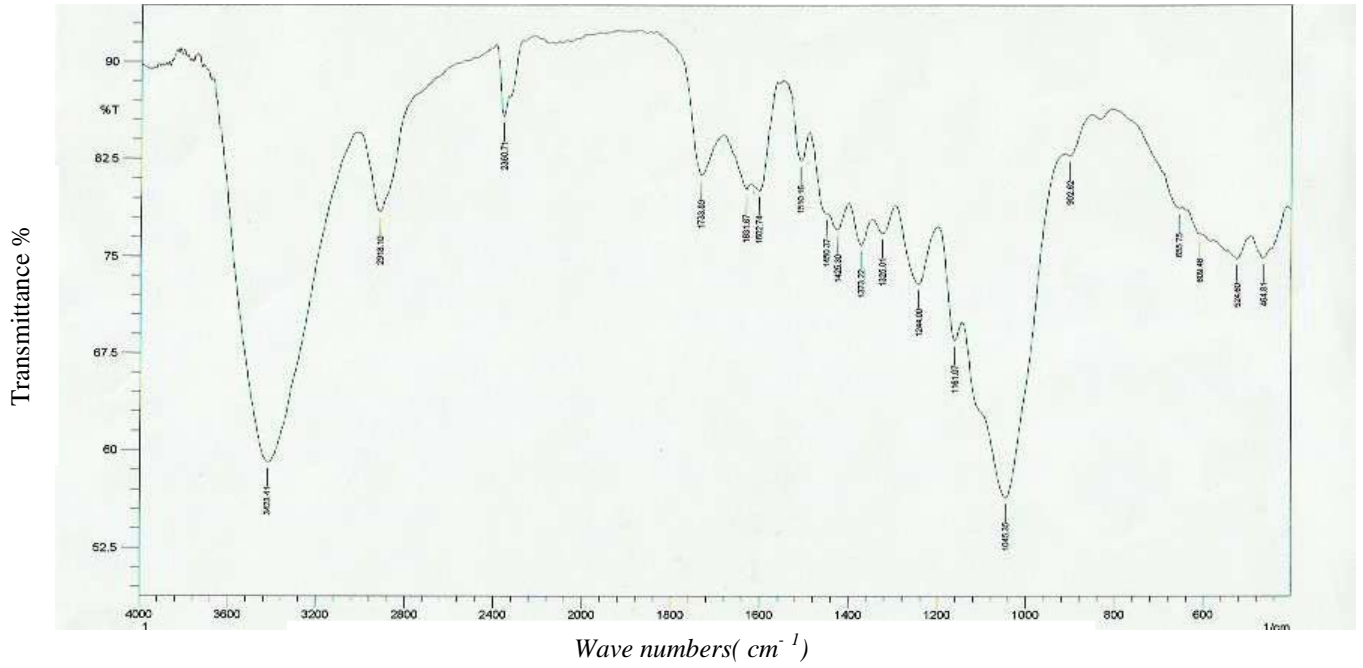
**Table (7):** IR patterns for Durra stalk

Peak of raw sample (Cm <sup>-1</sup> )	Transmittance %	Peak of α-cellulose (Cm <sup>-1</sup> )	Transmittance %	Peak of reference (Cm <sup>-1</sup> )	Transmittance %	Functional group
3419.56	54.8	3425.34	61	3368.59	17	OH stretching vibration
2921.96	57.2	2904.60	67.8	2901.11	31	C–H stretching vibration
1633.59	66	1635.52	68.9	1638.36	44.2	H <sub>2</sub> O absorption
1371.29	64.8	1371.29	66.3	1372.3	28.5	C–H bending
1061.13	53.2	1064.63	60.1	1058.76	14.1	C–O–C bending
898.77	70.5	896.84	67.5	896.52	32.1	C–O stretch& deformation
663.47	67.2	669.25	65.5	667.84	30.5	O–H bending

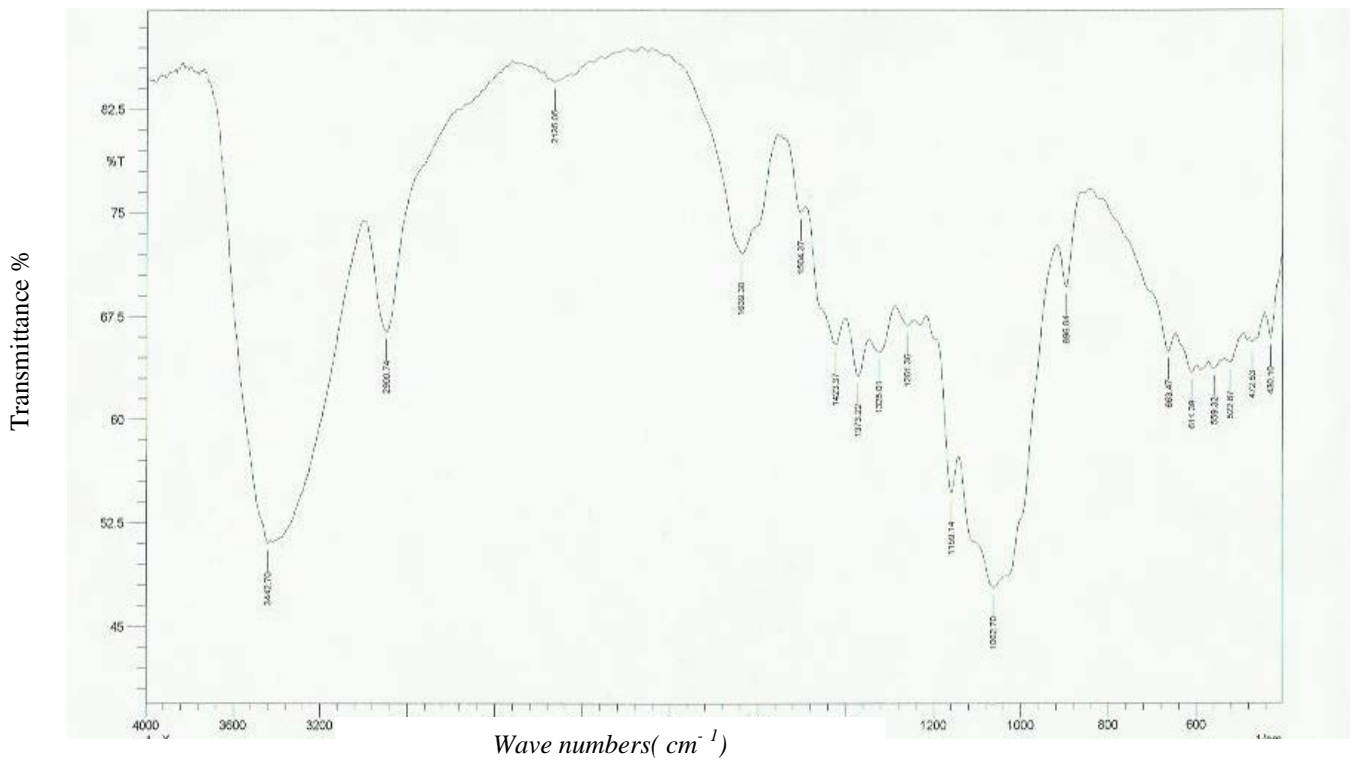
**Table (8):** IR patterns for Groundnut shell

Peak of raw sample (Cm <sup>-1</sup> )	Transmittance %	Peak of α- cellulose (Cm <sup>-1</sup> )	Transmittance %	Peak of reference (Cm <sup>-1</sup> )	Transmittance %	Functional group
3398.34	72.8	3431.13	46.8	3368.59	17	OH stretching vibration
2923.88	79.7	2908.45	63	2901.11	31	C–H stretching vibration
1647.10	80	1602.74	61.8	1638.36	44.2	H <sub>2</sub> O absorption
1371.29	81.4	1371.29	64.5	1372.3	28.5	C–H bending
1058.85	73	1031.85	53.4	1058.76	14.1	C–O–C bending
900.70	90	896.84	80.3	896.52	32.1	C–O str& deformation
630.68	87.8	663.47	78.8	667.84	30.5	O–H bending

Using the data shown by these tables and utilizing matlab software, a peak plots were performed and statistically compare the α-cellulose to the whole raw samples, figures (11) to (14) show that.

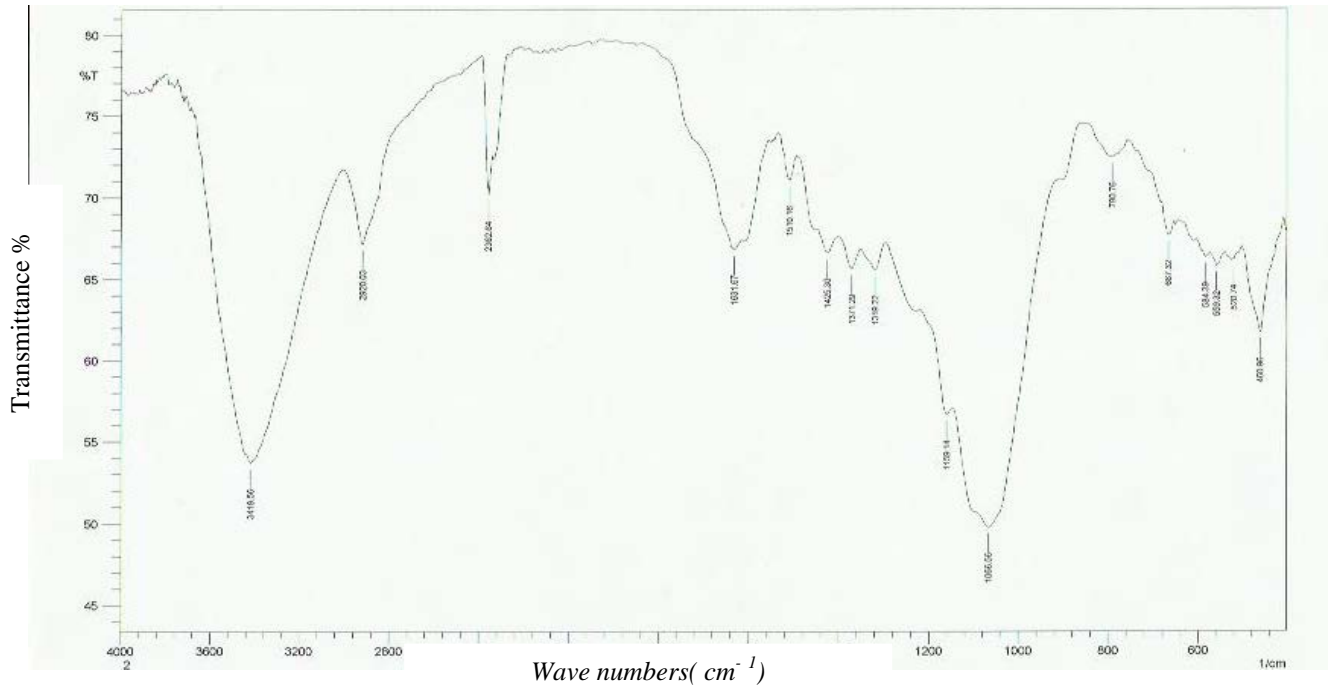


Fig(2) IR patterns for raw sugarcane bagasse

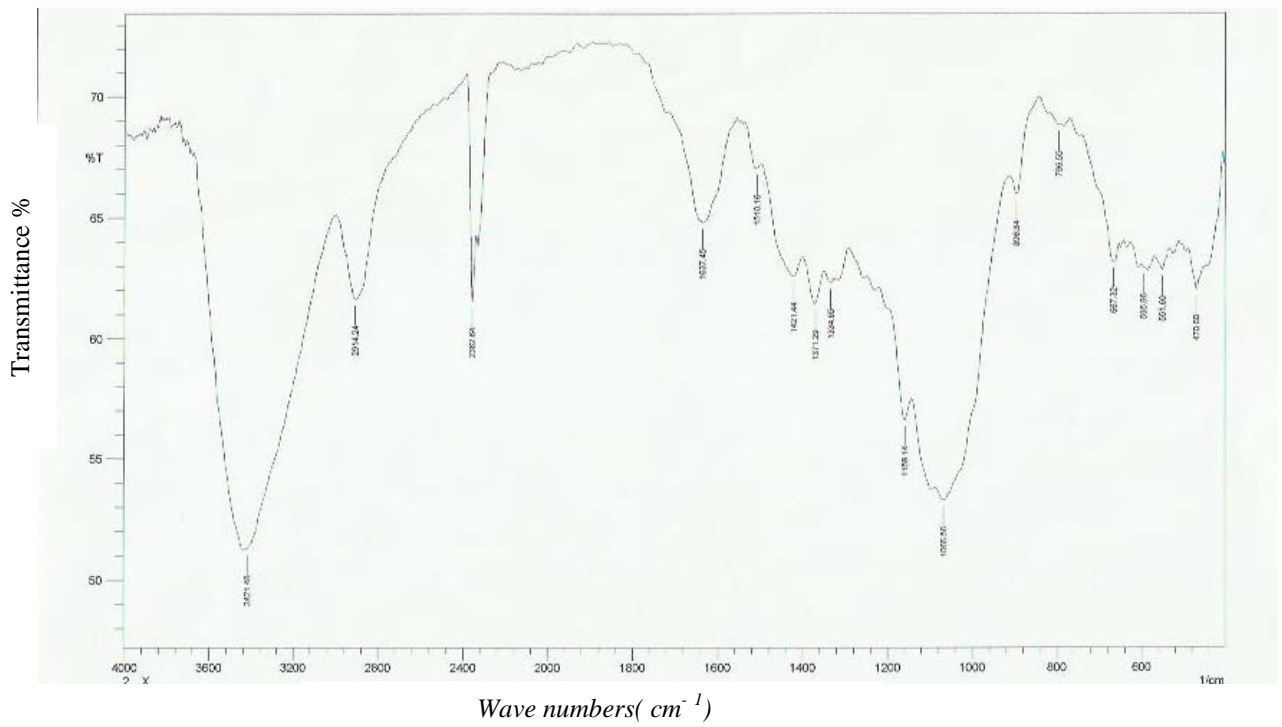


Fig(3) IR patterns for  $\alpha$ -cellulose from bagasse

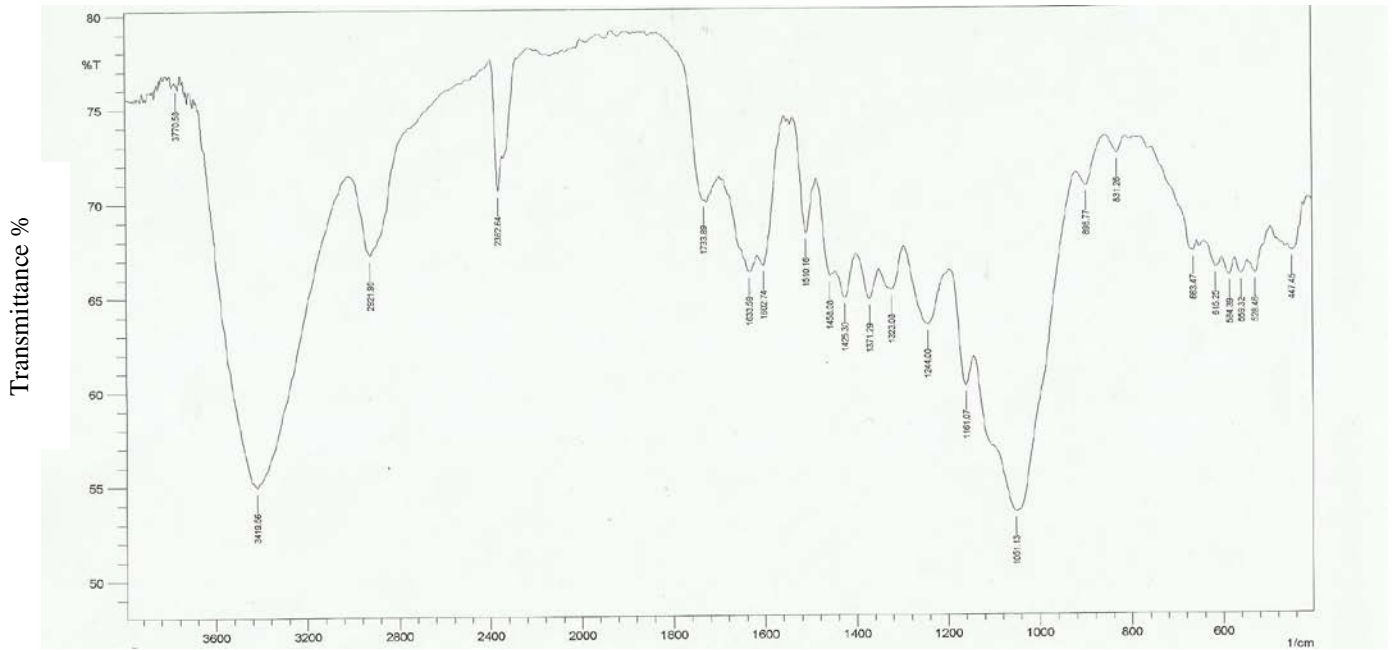




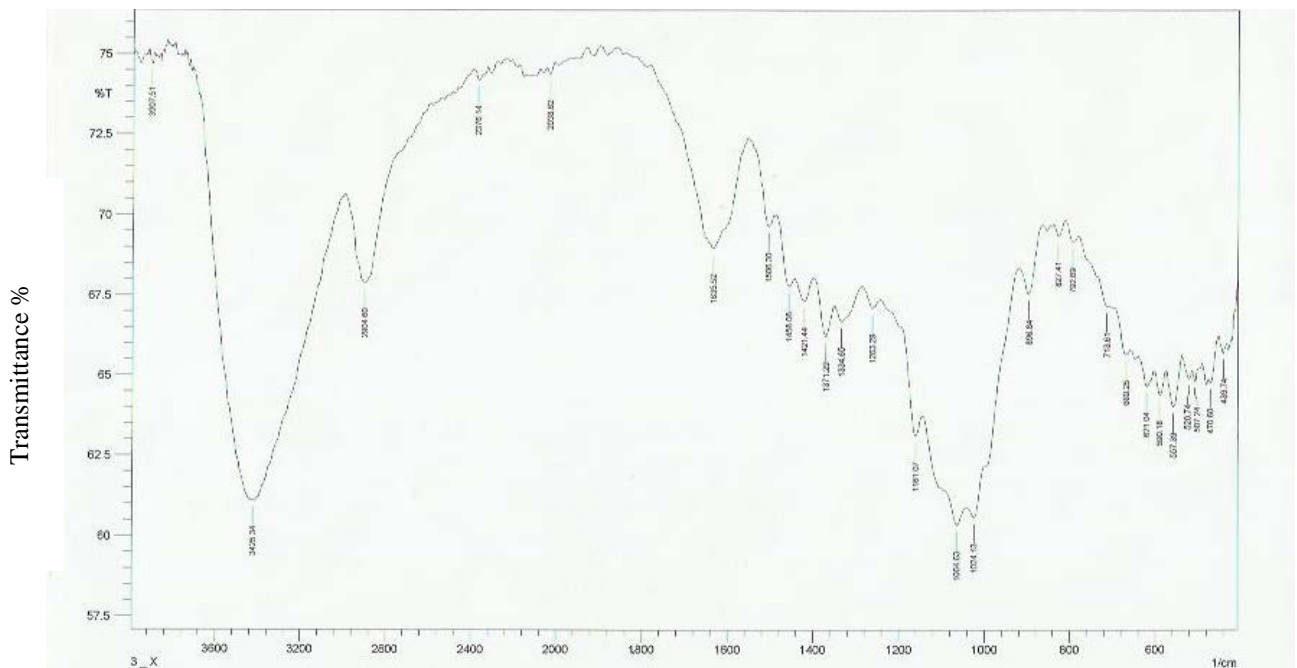
Fig(4) IR pattern for raw rice straw



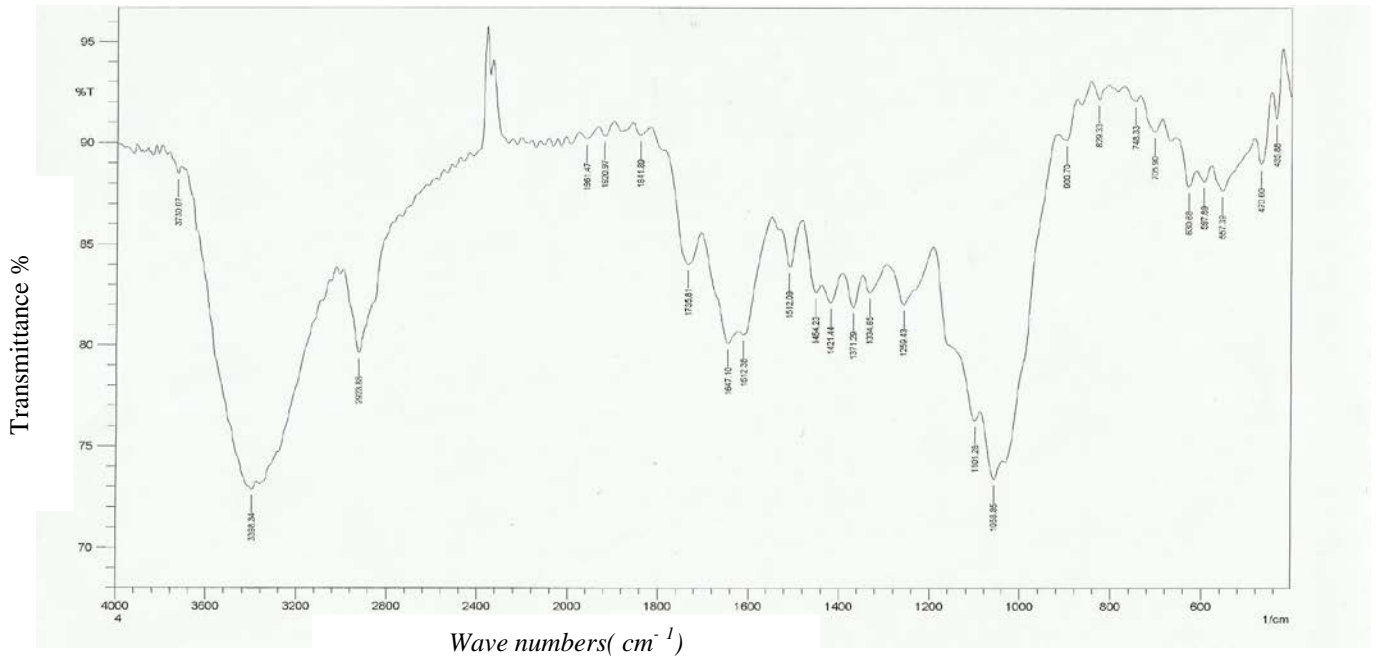
Fig(5) IR pattern for α- cellulose from rice straw



Wave numbers(  $cm^{-1}$  )  
Fig(6) IR pattern for raw durra stalk



Wave numbers(  $cm^{-1}$  )  
Fig(7) IR Patterns for  $\alpha$ - cellulose from durra stalk



Fig(8) IR patterns for raw groundnut shell

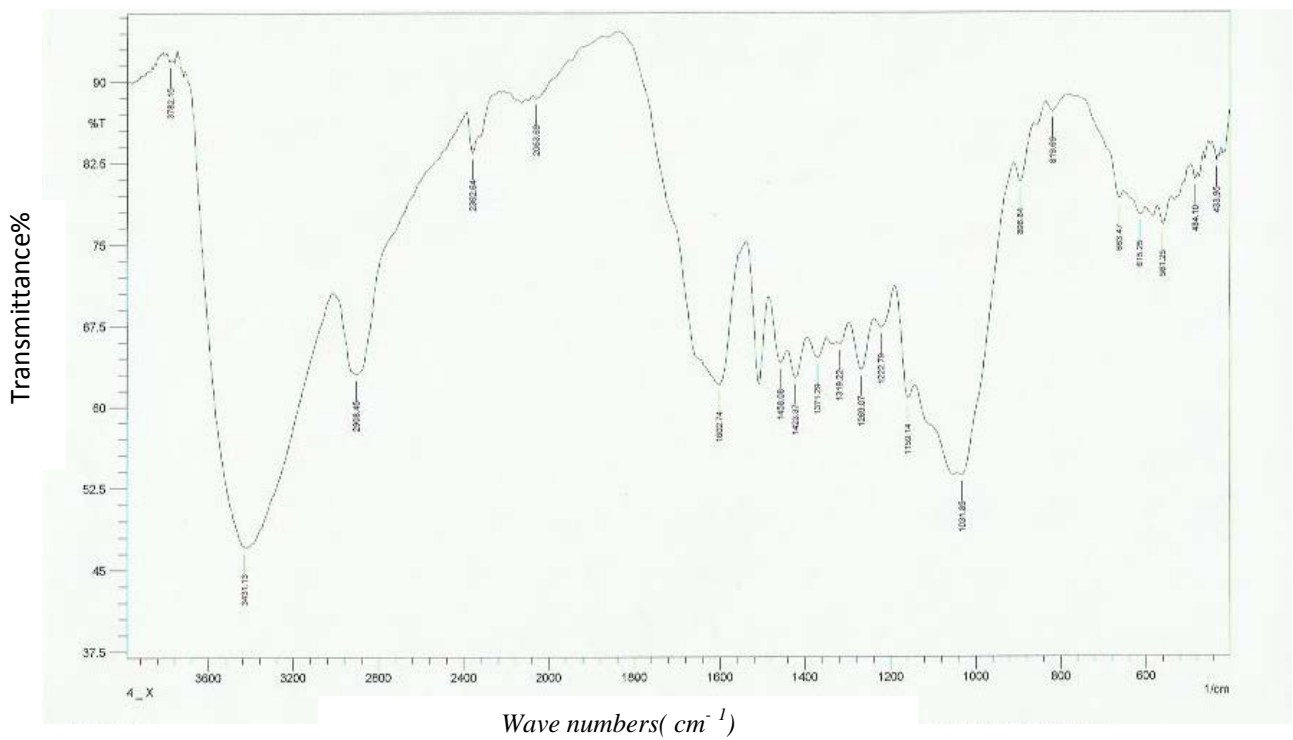


Fig (9) IR patterns for  $\alpha$ -cellulose groundnut shell

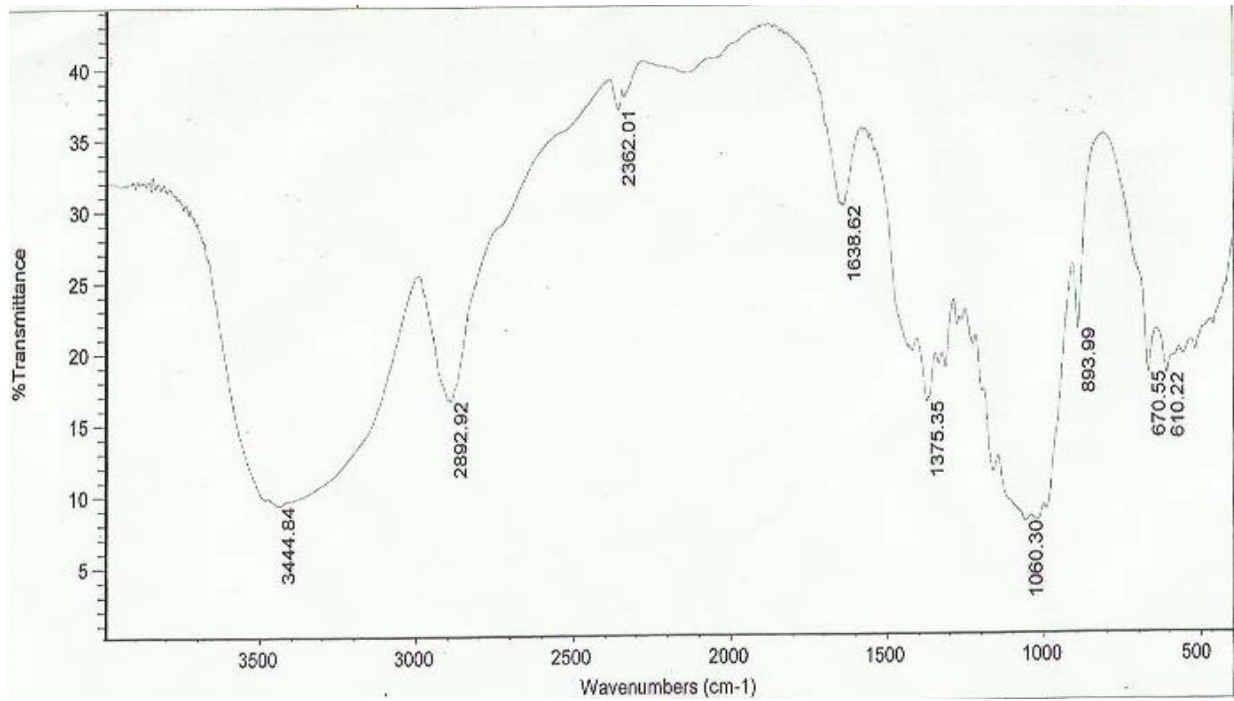


Figure (10): IR patterns for cellulose from medical cotton

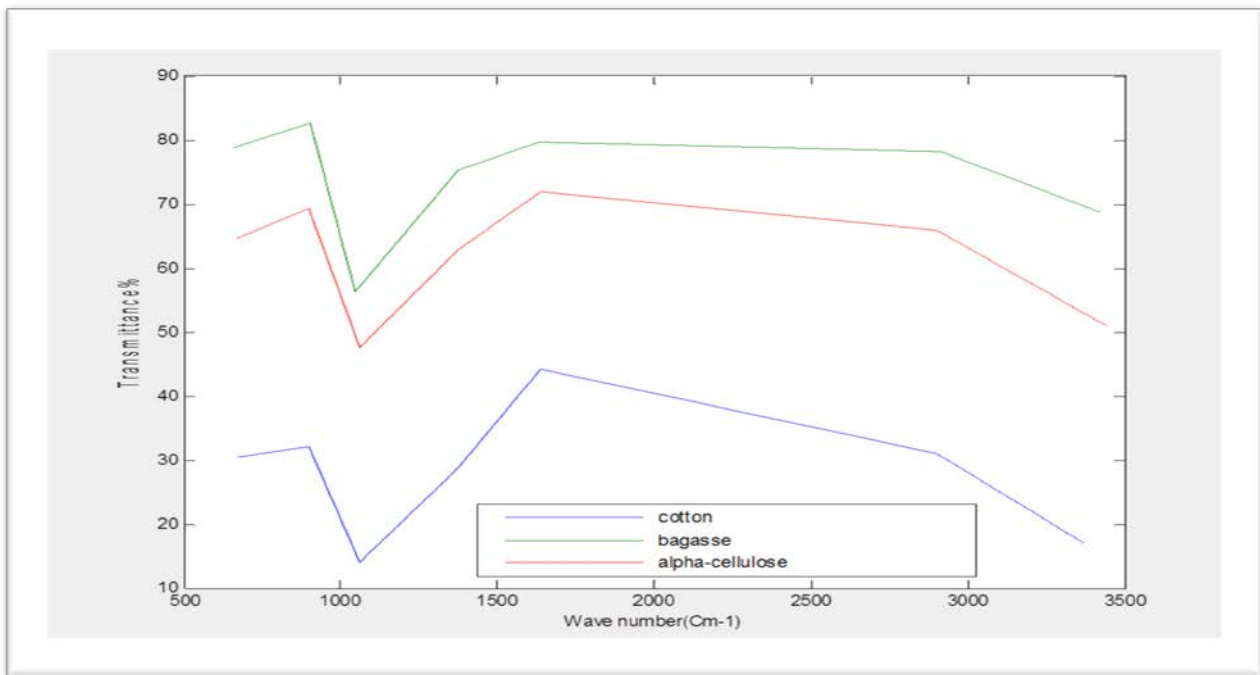


Fig (11) Schematic representation for data

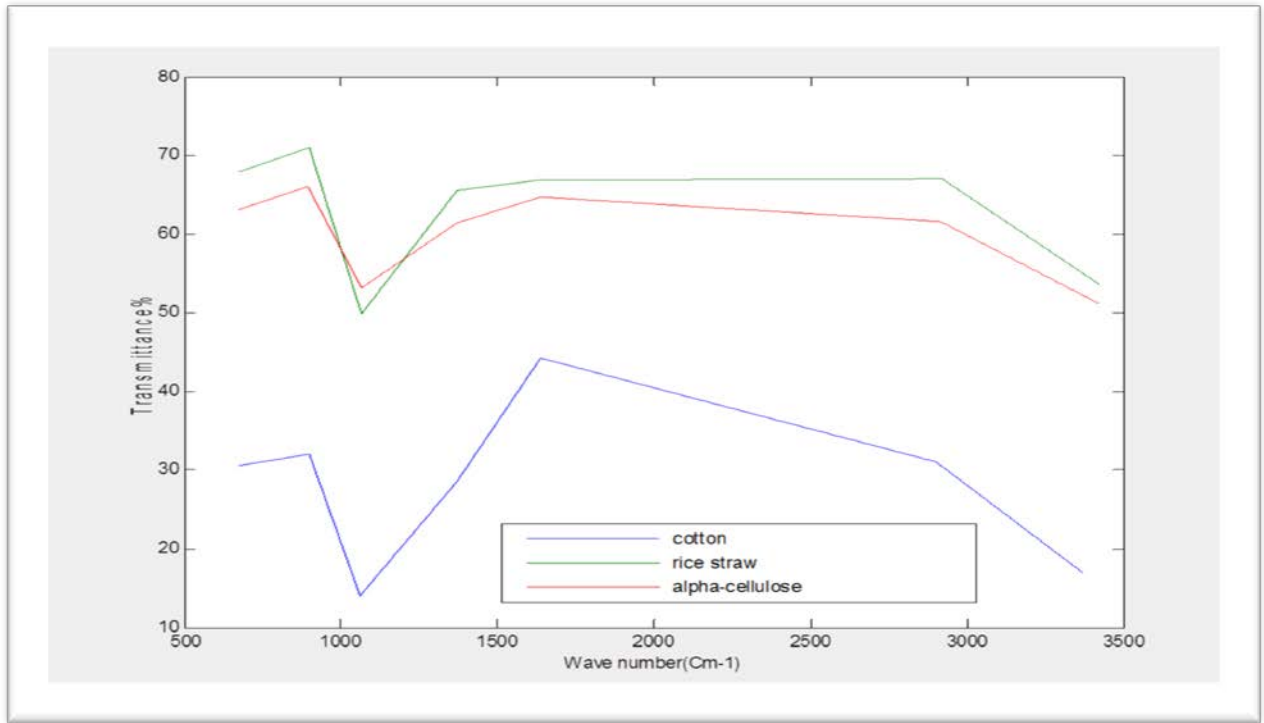


Table (12): Schematic representation for data of table(6)rice straw

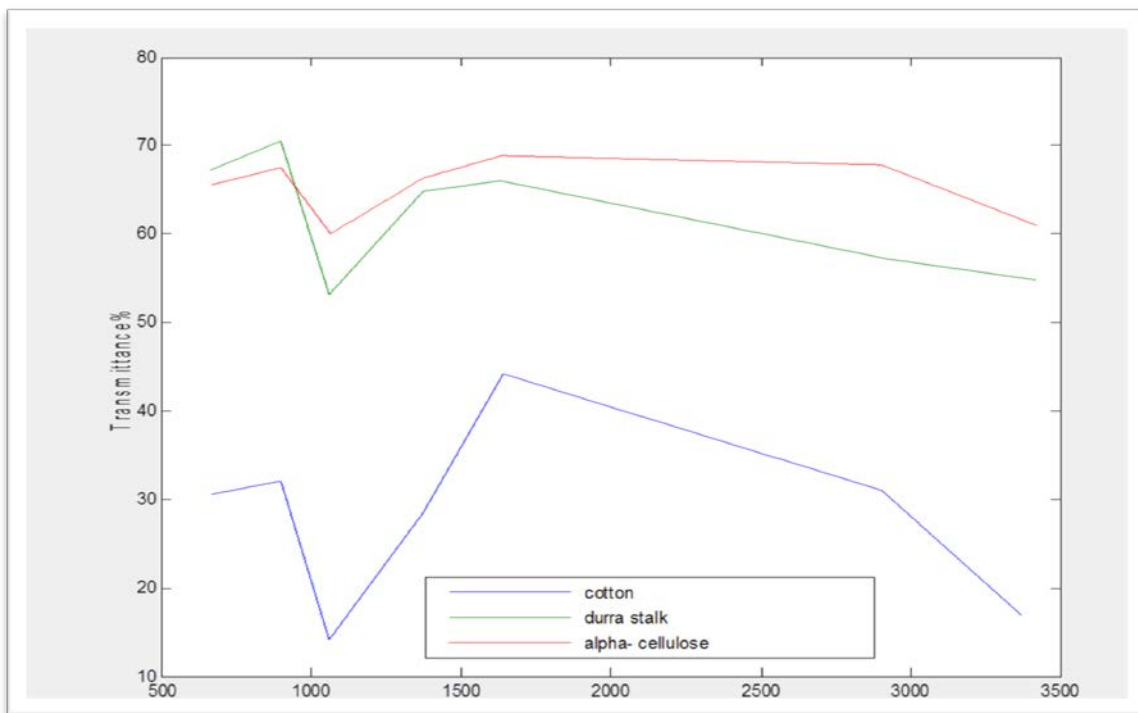


Fig (13) : Schematic representation for data of table (7)durra stalk

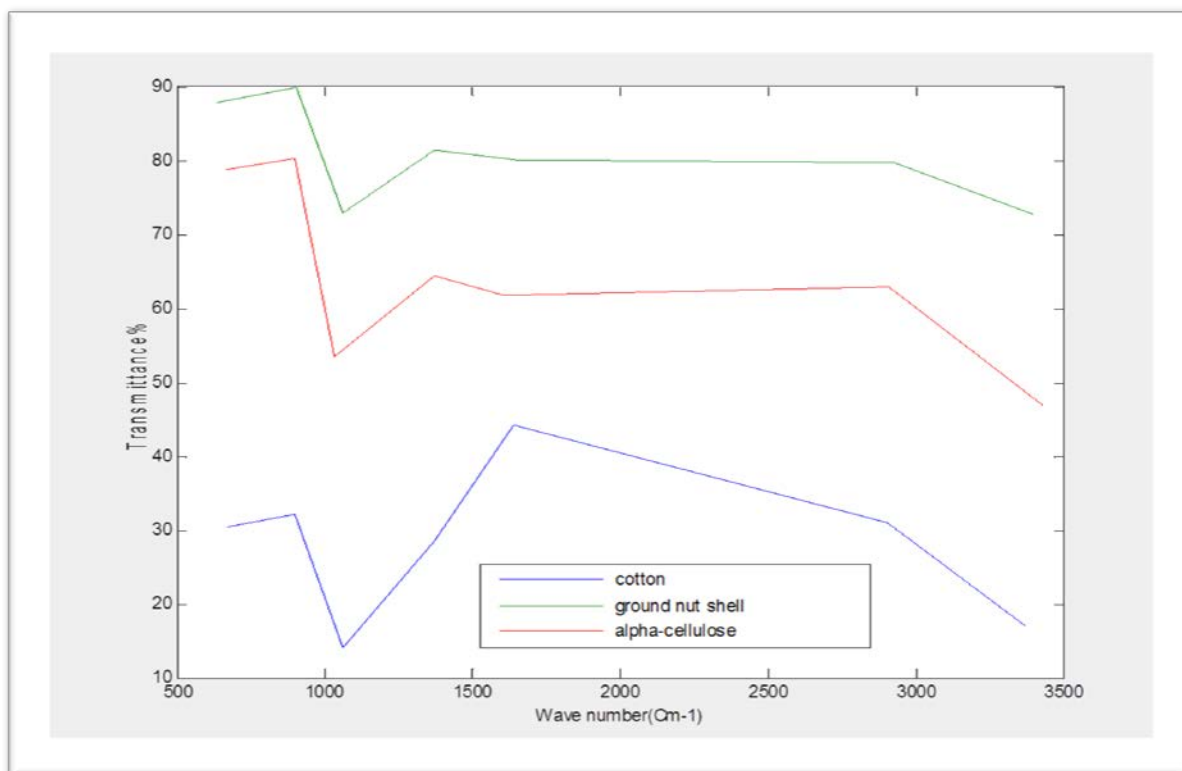


Fig (14): Schematic representation for data of table(8) groundnut shell

### 3.2. Discussions

- It may be tempting to use cellulose contents other than  $\alpha$ -cellulose to predict the actual content of the latter. However this study shows that no correlation exists between the total cellulose content in a source, or its content of alpha and its content of lignin (binder) or hemicelluloses or ash. It is noted from Tables (3 &4) and Fig.(1) that all four of the materials tested have about the same value of total cellulose content, yet groundnut shell stands out with a higher  $\alpha$ -cellulose content. It is also noted that the ash content of groundnut shell is significantly high, yet this did not deter this material from having higher  $\alpha$ -cellulose than the other three sources. The important lesson learned is that total cellulose content of a plant source, or the content of lignin or ash content, cannot be used as dependable criteria for judging the usefulness of a plant source for nitrocellulose production.
- The values obtained for  $\alpha$ -cellulose, are rather lower than those reported by other researchers. Both sets of data are limited; are the values obtained in this study too low or are the others too high, given that the methods used are quite similar. These methods involve a number of sources of error, notably:
  - Useful cellulose may be removed with non-cellulose ingredients, during the initial extraction process.
  - The delignification process can remove pure cellulose with lignin by entrapment or otherwise.

- The most probable source of error could be isolation of  $\alpha$ -cellulose from hemicelluloses, given the similarity of chemical nature.
- In comparing the IR spectra obtained on the four plant sources and that obtained on pure cotton cellulose (used as reference), very close match is observed, the discrepancy does not exceed ~ 0.022%. Probably, the most important observation on these spectra is the presence of absorption peak at (3000-3500), which is characteristic of OH group. This group plays an important role during the nitration reaction for production of nitrocellulose.

#### 4. Conclusion and Recommendations

##### 4.1. Conclusions

- Of the four materials investigated, groundnut shell is shown to be the best source of  $\alpha$ -cellulose.
- Previous studies have shown cotton fibers to yield the highest  $\alpha$ -cellulose content, ~ 96%. However, cost-effectiveness hinders this source of  $\alpha$ -cellulose from being highly attractive, less expensive sources, especially local ones need to be identified. Only four sources of agricultural wastes have been investigated in this study, a more extensive list need to be investigated.
- The study has shown *liquid-solid extraction* and *alkaline hydrolysis* to be suitable methods for the isolation of  $\alpha$ -cellulose from plant sources. However, the same cannot be said about the materials employed in the various processes.

##### 4.2. Recommendation

- The present study has been limited to only four sources of  $\alpha$ -cellulose, all of which are cheap materials (agricultural residue). A larger database need to be built covering local inexpensive sources of cellulose.
- The materials used in this , and other studies, are limited (2:1 ethanol-toluene, acidic Na-chlorite, 18% NaOH). The effectiveness of these formulations has not been systematically determined.
- Per se, we cannot say that a certain material is suitable for  $\alpha$ -cellulose production, unless we specify the environment of the plant production. Groundnut shell produced in semi-arid, sandy dessert of northern Kordofan state may not be the same as the waste produced in clay soil of Gezira State.
- The IR peak detected at (3000-3500) may be very useful in controlling the degree of substitution during nitration. This is an important parameter which determines the type of product obtained, and its control has been rather elusive.

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