

**RESEARCH ARTICLE****Investigation Studies of Microwave Effect on Structure of Cellulosic Fibers****Samar H. Mohamed¹, Monazah G. Khafagi², Zenat A. Nagieb¹ and M. A. El-Samahy³**¹Cellulose and Paper Department, National Research Center²Spectroscopic Department, National Research Center³Packaging Department, National Research Center**Manuscript Info****Manuscript History:**

Received: 12 April 2014
Final Accepted: 23 May 2014
Published Online: June 2014

Key words

Paper sheets, Infra red spectroscopy, Scanning electron microscopy, Strength properties, Optical properties

Corresponding Author*Samar H. Mohamed****Abstract**

Different types of fiber were used to prepare paper. The produced paper sheets were submitted to accelerate ageing at 100°C for 72hr. It is said that 72hr. would be equal to 25 years under ambient conditions and also we used microwave for comparison and investigate microwave effect on structure of Cellulosic fibers. Paper obtained from cotton pulp retained its original properties with time, paper obtained from bagasse and wood pulps gave a marked improvement in mechanical properties with time comparing with untreated. Mechanical properties of paper prepared from unbleached and bleached rice straw deteriorated with time comparing with untreated. In all cases whiteness affected by submitting paper sheets to accelerate ageing, but this observed clearly in case of paper sheets obtained from unbleached pulps. From scanning electron microscopy, it had been observed that, paper sheets obtained from bagasse (bleached and unbleached) and wood pulps which submitted to accelerate ageing at 100°C for 72 hr. and also by using microwave presented certain adhesion between the fibers comparing with untreated sheets. Also, from infrared spectroscopy, it had been observed that, in case of paper sheets obtained from bagasse (bleached or unbleached) and wood pulps, crystallinity index increased when paper sheets submitted to accelerate ageing comparing with untreated sheets.

*Copy Right, IJAR, 2014. All rights reserved.***INTRODUCTION**

Paper is a thin material mainly used for writing upon, printing upon or for packaging. It is produced by pressing together moist fibers, typically cellulose pulp derived from wood, rags or grasses, and drying them into flexible sheets. The use of paper spread from China through the Islamic world and entered production in medieval Europe in the 13th century, where the first water-powered paper mills were built and mechanization of papermaking began. The industrial production of paper in the early 19th century caused significant cultural changes worldwide, allowing for relatively cheap exchange of information in the form of letters, newspapers and books for the first time. In 1844, both Canadian inventor Charles Fenerty and German inventor F.G.Keller had invented the machine and process for pulping wood for the use in paper making (Burger, 2007). This would end the nearly 2000-year use of pulped rags and start a new era for the production of newsprint and eventually all paper out of pulped wood.

The factor that determines the ageing behavior of a paper is how it was manufactured, not the original source of the fibres (Erhardt and Tumosa, 2005). Furthermore tests sponsored by the Library of Congress prove that all paper is at risk of acid decay, because cellulose itself produces formic, acetic, lactic and oxalic acids.

Permanent paper is paper that have a reasonable life expectancy exceeding 200 year. Most modern papers have a reasonable life expectancy of about 50 year (Houssni et al., 1998), but durability is the ability of paper to keep its

original characteristics in use. Archival paper is an especially permanent, durable acid-free paper. Archival paper is meant to be used for publications of high legal, historical, or significant value. According to the scope of the standard, archival paper is primarily required for documents and publications intended to be kept permanently because of their high historical, legal or other significant values. Archival paper is for special purposes, not for common use. The use of the term "archival paper" does not imply that all papers kept in archives are "archival papers" (Ivar and Hoel, 2009; Dahlo, 2000)

The best method of measuring the permanence of paper is by natural aging. However, this method is obviously impractical for relatively permanent papers because of the time involved. In order to shorten the time involved, accelerated test methods have been devised which utilize light and heat (Shahani, 1998 ; Stroofer, 1990). Heat tests may be used to measure the relative stability of paper, although heating does not always give results comparable to natural aging because of the dehydration which occurs at high temperatures. In carrying out accelerated aging tests using heat, paper strips are heated in an oven at 105°C. for seventy-two hours and then tested for folding endurance (Altaf et al., 2006).

Also, it can be seen that heating by conventional means is a slow process, involving a number of stages in the transfer of energy before the material to be heat reaches a uniform state of molecular activity and temperature. The property of microwaves, which makes them attractive for dye- fixation and other uses, is their ability, under suitable conditions, to produce rapid and uniform heating throughout the material exposed to them (Motasemi and Afzal, 2013; Gawande et al., 2014; Ravoof et al., 2012).

The aim of the work is to investigate microwave effect on structure of Cellulosic fibers and also heating by conventional means. Mechanical and optical properties of paper sheets were studied. Also scanning electron microscopy is very important, and The determination of the crystallinity of polymer is a very important industrial application of infrared spectroscopy. Infrared method is rapid and convenient.

Experimental

Materials

Bleached and unbleached rice straw, cotton, bleached and unbleached bagasse, unbleached mixed (rice straw and 25% bagass), and wood pulps, were used as a base furnish to prepare papers. supplied by Edfo Paper Mill, Upper Egypt.

Paper making

The pulps were beaten in a Jokro beater at 6% consistency until they reached 45°SR. After paper sheet formation, the papers were conditioned for 24 hr., at 50% RH and 20°C (El-Saied et al., 2000; Basta, 2003). The pulps were chemically analyzed for α -Cellulose, pentosans, lignin, and ash, according to standard methods (Basta, 2004).

Accelerated Ageing

The produced paper sheets were submitted to accelerate ageing at 100°C for 72hr. It is said that 72hr. would be equal to 25 years under ambient conditions (Altaf et al., 2006; Rozmarin, 1961; Kirova, 1977) and also we used microwave 2hr. at 270 watt (Breitwieser et al., 2013; Aiqin and Lianghua, 2008). Sheets had been subjected to the following tests:

Mechanical properties

Mechanical properties of the paper, namely tensile strength, breaking length and burst strength had been measured (Casey, 1981).

Physical Properties

Physical property of the produced paper sheets namely whiteness had been measured by using Ultra Scan Pro Hunter lab(w₁ E13[D65/10]).

Chemical analysis of paper sheets

Most of the analytical methods used in this work were carried out according to the German standard (Samar, 1996).

Infrared spectra

The infrared spectra for treated and untreated prepared papers fiber were measured by using Fourier transform infrared spectrometer (FT/IR-6100, Jasco, Japan). All spectra were recorded in the range (4000 – 400 cm⁻¹), the number of scans was 128, and the resolution was 4 cm⁻¹ and scan speed 2 mm /s. Infra-red spectra of the papers fiber were obtained in reflection % to analyze the chemical composition.

Scanning electron microscopy:

Scanning electron microscopy (SEM) were conducted on JEDL JEM-100 S electron microscope using the gold – Sputtering technique.

Results and Discussion

The chemical analysis of raw materials are given in table (1).

Mechanical and physical properties of untreated paper sheets.

Table (2) shows mechanical(tensile strength, breaking length and burst strength) and physical properties(whiteness%) of untreated paper sheets which prepared from (bleached and unbleached rice straw, cotton, bleached and unbleached bagasse, unbleached mixed (rice straw and 25% bagass), and wood) pulps. From this table, it is clear that, paper sheets prepared from bagasse and wood pulps gave best results in mechanical properties, this can be attributed to the different chemical composition of pulps (Samar, 1996). But in case of physical properties (whiteness%) paper sheets prepared from cotton pulp gave best results.

Mechanical and physical properties of paper sheets submitted to accelerate ageing at 100°C for 72hr., and also by using microwave(2hr. at 270watt)

From tables (3,4) it is clear that the paper obtained from bagasse pulp showed an improvement in the mechanical properties of the paper after heating, this can be attributed to the different chemical composition of pulps. From table(1) it is clear that, the pentosans and lignin content are higher in the bagasse pulp. These pentosans are softened by heating, which enhance adhesion of the fibers together, and is responsible for the observed improvement in the tensile and burst strength of the paper (Samar, 1996). Also paper sheets prepared from wood pulp showed an improvement in the tensile strength for the same reason. From tables (3,4) it is clear that, mechanical properties of paper prepared from unbleached and bleached rice straw deteriorated by submitting paper sheets to accelerate ageing at 100°C for 72hr., and also by using microwave (2hr. at 270watt), but there is no change in case of paper sheets prepared from mixed and cotton pulps. In all cases whiteness affected by submitting paper sheets to accelerate ageing, but this observed clearly in case of paper sheets obtained from unbleached pulps which contain lignin. Lignin is particularly sensitive to light and undergoes a photochemical reaction in sunlight leading to a darkening of the lignin.

Scanning electron microscopy of different samples

Photographs of Fig.1(b,c) comparing with Fig.(1a) it is clear that paper sheets obtained from unbleached bagasse pulp which submitted to accelerate ageing at 100°C for 72 hr. and also by using microwave present certain adhesion between the fibers. From table (1) it is clear that the pentosans and lignin content are higher in the bagasse pulp. These pentosans are softened by heating, which enhance adhesion of the fibers together, and has more compact structure than untreated sheets, showing a cleaner pulp with no of fibrillation or filaments between the fibers no cracks or void thus the crack does not have a straight path because it has to move around the fiber cells & ultimately stops (Li, 2003). Also, in fig.1(e,f,k,l) paper sheets obtained from bleached bagasse and wood pulps which submitted to accelerate ageing at 100°C for 72 hr. and also by using microwave present certain adhesion between the fibers comparing with untreated sheets fig.1(d,j). But this is not occur in fig.1(g,h,i). This is attributed to chemical analysis of pulps (table 1).

Infrared spectroscopy

Table (5) shows the ratio $A_{1435\text{ cm}^{-1}}/A_{900\text{ cm}^{-1}}$ which represents the crystallinity index for the paper sheets made from different pulps. crystallinity index decreases in case of paper obtained from rice straw pulp (bleached or unbleached), when paper sheets submitted to accelerate ageing at 100°C for 72hr. and also when paper sheets submitted to accelerate ageing using microwave (2hr. at 270watt) this is related to components of cellulosic or lignocellulosic materials in paper sheets, indicating that, paper sheets contain less hydrogen bonds between the hydroxyl groups in cellulosic chain. In case of sheets obtained from mixed and cotton pulps, there is no clearly change in crystallinity index when paper sheets submitted to accelerate ageing at 100°C for 72hr. and also when paper sheets submitted to accelerate ageing using microwave. But in case of paper sheets obtained from bagasse (bleached or unbleached) and wood pulps, crystallinity index increases when paper sheets submitted to accelerate ageing at 100°C for 72hr. and also when paper sheets submitted to accelerate ageing using microwave comparing with untreated sample, this is related to components of cellulosic or lignocellulosic materials in paper sheets, indicating that, paper sheets contain more hydrogen bonds between the hydroxyl groups in cellulosic chain (Samar, 1996).

Table (1): Chemical analysis of different types of pulps.

Type of pulp	Lignin %	Pentosan %	Alpha Cellulose %	Ash %
Bleached rice straw	0.9	22.5	67.0	1.4
Unbleached rice straw	5.6	23.9	68.4	1.6
Cotton	0.5	1.5	96	1.0
Unbleached bagasse	5.4	25.6	69.0	0.9
Bleached bagasse	0.9	24.0	67.2	0.8
Mixed (rice straw and 25% bagasse)unbleached	1.9	23.9	72.5	1.6
Wood	traces	9.0	89.5	0.5

Table (2): Mechanical and physical properties of untreated paper sheets.

Type of paper	Mechanical properties			Physical properties
	Tensile strength, kg	Breaking length, m	Burst strength, kg/ Cm ²	Whiteness %
Paper made from unbleached rice straw pulp	0.9	882.353	0.4	15.47
Paper made from bleached rice straw pulp	0.3	294.118	0.2	40.67
Paper made from unbleached bagasse pulp	2.500	2450.980	1.0	25.89

Paper made from bleached bagasse pulp	2.250	2205.882	0.9	43.39
Paper made from unbleached mixed (rice straw and 25% bagasse) pulp	1.700	1666.666	0.8	14.51
Paper made from Cotton pulp	1.300	1274.510	0.6	60.51
Paper made from Wood pulp	3.500	3431.373	2.6	53.70

Table (3): Mechanical and physical properties of paper sheets submitted to accelerate ageing at 100°C for 72hr.

Type of paper	Mechanical properties			Physical properties
	Tensile strength, kg	Breaking length, m	Burst strength, kg/ Cm ²	Whiteness %
Paper made from unbleached rice straw pulp	0.65	637.255	0.3	13.71
Paper made from bleached rice straw pulp	0.35	343.137	0.3	36.02
Paper made from unbleached bagasse pulp	2.54	2490.196	1.3	20.33
Paper made from bleached bagasse pulp	2.01	1970.588	0.9	40.57

Paper made from mixed (rice straw and 25% bagasse) unbleached pulp	1.36	1333.333	0.6	11.70
Paper made from cotton pulp	1.10	1078.431	0.6	50.72
Paper made from wood pulp	3.94	3862.745	2.2	50.76

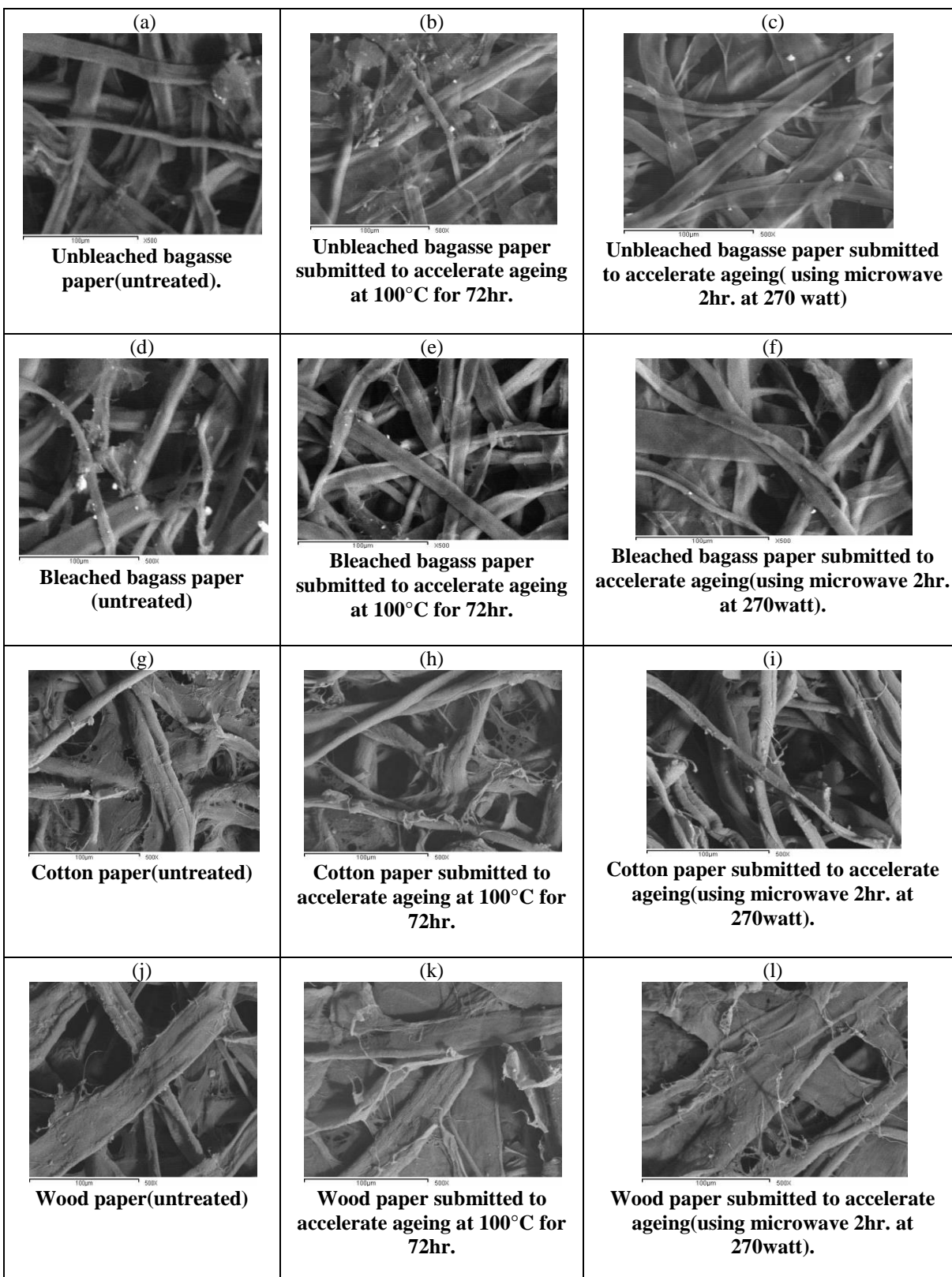
Table (4): Mechanical and physical properties of paper sheets submitted to accelerate ageing using microwave (2hr.at 270watt).

Type of paper	Mechanical properties			Physical properties
	Tensile strength, kg	Breaking length, m	Burst strength, kg/ Cm ²	Whiteness %
Paper made from unbleached rice straw pulp	0.70	686.275	0.3	12.19
Paper made from bleached rice straw pulp	0.24	235.294	0.2	35.11
Paper made from unbleached bagasse pulp	2.580	2529.412	1.2	19.51
Paper made from bleached bagasse pulp	1.710	1676.471	1.0	41.59
Paper made from mixed (rice straw and 25% bagasse) pulp	1.300	1274.510	0.6	10.76
Paper made from cotton pulp	1.020	1000	0.5	49.85

Paper made from wood pulp	3.900	3823.529	2.5	50.06
---------------------------	-------	----------	-----	-------

Table (5): The ratio $A_{1435\text{cm}^{-1}}/A_{900\text{cm}^{-1}}$ which represents the crystallinity indexes for paper sheets made from different pulps

Type of paper	Untreated paper	Treatment of paper at 100°C for 72hr.	Treatment of paper using microwave (2 hr. at 270 watt)
Paper made from unbleached rice straw pulp	0.983	0.777	0.767
Paper made from bleached rice straw pulp	1.065	1.002	0.974
Paper made from unbleached bagasse pulp	0.650	0.815	0.653
Paper made from bleached bagasse pulp	0.935	1.023	1.237
Paper made from unbleached mixed (rice straw and 25% bagasse) pulp	1.098	1.158	1.006
Paper made from cotton pulp	2.303	2.411	2.509
Paper made from wood pulp	1.312	1.315	1.677

Figure (1) Scanning electron microscopy of different samples (a-l).

Conclusion

Paper sheets prepared from bagasse and wood pulps gave marked increase in mechanical properties than untreated when submitting paper sheets to accelerate ageing at 100°C for 72hr., and also by using microwave (2hr. at 270watt), this can be attributed to the different chemical composition of pulps. But in case of physical properties (whiteness %), paper sheets prepared from cotton pulp gave best results. Mechanical properties of paper prepared from unbleached and bleached rice straw deteriorated by submitting paper sheets to accelerate ageing at 100°C for 72hr. and also by using microwave, but there is no change in case of paper sheets prepared from mixed and cotton pulps. In all cases whiteness affected by submitting paper sheets to accelerate ageing at 100°C for 72 hr. and also by using microwave, but this observed clearly in case of paper sheets obtained from unbleached pulps which contain lignin. Scanning electron microscopy proved all of the above. Also from infrared spectra, the ratio A_{1435} / A_{900} represents the crystallinity index for paper sheets made from different pulps, it has been observed that, in case of paper sheets obtained from bagasse (bleached or unbleached) and wood pulps, crystallinity index increases when paper sheets submitted to accelerate ageing at 100°C for 72hr. and also when paper sheets submitted to accelerate ageing using microwave comparing with untreated sample.

References

- Aiqin HouXiaojun Wang, Lianghua Wu (2008) Effect of microwave irradiation on the physical properties and morphological structures of cotton cellulose74, Pages 934–937.
- Altaf H., Hossni E., Samar H. and Samya E. (2006) The role of neutral rosin- alum size agent in production of permanent Paper. *Restaurator* 27, P.67-80.
- Basta, A. H. (2003) The role of chitosan in improving the ageing resistance of rosin sized paper. *Restaurator* 24, 106.
- Basta, A.H. (2004) Performance of improved polyvinyl alcohol as ageing resistance agent of rosin sized paper and in restoration purpose. *Restaurator* 25, 129-140.
- Breitwieser D, Moghaddam MM, Spirk S, Baghbanzadeh M, Pivec T, Fasl H, Ribitsch V, Kappe CO. (2013) In situ preparation of silver nanocomposites on cellulosic fibers –Microwave vs. conventional heating. *Carbohydrate Polymers* 94, 677– 686.
- Burger,P. Charles. Fenerty and his Paper Invention. (2007) Toronto: PeterBurger, ISBN978-0-9783318-1-8 P.25-30.
- Casey, J. P. (1981) Paper testing and converting in pulp and paper (3rd ed.). New Yourk Interscience Publisher pp. 1714–1965.
- Dahlo R. (2000) The Rationle of Permanent Paper. In W. Manning&V. Kremp(Eds.) IFLA Publications 91: A Reader in preservation and Conservation. 58. Munchen:K.G.Saur.
- El-Saied H., A.H. Basta & M.M. Abdou (2000) Permanence of paper II: Correlation between paper permanence from straw and ageing variables. *Restaurator* 21: 158.
- Erhardt D., Tumosa C. (2005) Chemical Degradation of Cellulose in Paper over 500years. *Restaurator* 26,155.
- Gawande M. B., S. N. Shelke, R. Zboril and R. S. Varma (2014) Microwave-Assisted Chemistry: Synthetic Applications for Rapid Assembly of Nanomaterials and Organics, *ACC. CHEM. RES.*, vol. 47, iss. 4, pp. 1338-1348.
- Houssni E., Altaf H. and Mona M.(1998) Permanence of Paper 1. Problems and permanency of Alum–Rosin Sized Paper Sheets from Wood pulp. *Restaurator* p.155-171.

Ivar A. and Hoel I. (2009) Standards for permanent Paper. 64th IFLA general Conference Programme and Proceedings. Archive . ifla. Org. Retrieved, 06, 24.

Kirova,Kn.L.,I.A.Stepanova, D.M.Flyate &I.S. Shul-man(1977) (Kinetic studies in the ageing of papyrus). Problemy Sokhraunosti Documen-talnykh Materialov. Leningrad 13.

Li, Q.; LM. Mutua, N. J. (2003) Appl. Polym. Sci 88, 278.

Motasemi, F., Afzal, M.T.(2013) A review On the microwave assisted pyrolysis technique. Renov. Sustain. Energy Rev., **28**,317-330.

Ravoof S. A., K. Prateepa, T. Supassri, and S. Chittibabu (2012) “Enhancing enzymatic hydrolysis of rice straw using microwave-assisted nitric acid pretreatment,” International Journal of Medicine and Biosciences, vol. 1, no. 3, pp. 13–17.

Rozmarin, Gh. (1961) Ageing of fibrous cellulose materials. Cellulza si Hirtie 10:428.

Samar H. Mohamed, M.Sc. (1996) Improving physical and mechanical properties of paper sheets by adding some additives , Benha University.

Shahani C., Hengemihle H. and Weberg N. (1989) The Effect of Variations in Relative Humidity on the Accelerated Aging of Paper. American Chemical Society P. 63-79.

Stroefer H. (1990) Experimental Measurement: Interpreting, Extrapolation and Prediction by Accelerated Aging. Restaurator 11,4, P. 254-266.