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Improving the Wet Tensile Strength of Recycled Paper via Incresing its Bond Capacity

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Abstract: This research relates to treated deinked Mixed Office Waste (MOW) paper cellulosic fibers and is directed particularity to a method for improving the wet strength of recycled paper. The effect of chemical treatment of fiber was investigated. Fibers treated by 6 and 10% Succinic Acid (SA) respectively with 3 and 5% (half-one of carboxylating agent) catalysts (based on the total weight of fibers). The catalysts used for this research were Sodium dihydrogen Phosphate (SDHP) and Sodium hypophosphite (SHP). On the other hand, 0 and 1% cationic polymeric resin or Anionic Trash Catcher (ATC) and 2% wet strength resin or Poly Aminoamide-Epichlorohydrin (PAE) was used to treated fibers. All of handsheets plus control handsheet were treated with 2% PAE. Also, chemical structure of treated fibers with Succinic Acid and catalysts Only and in two pH, was investigated by XRD and FTIR analyses. Results of these treatments showed that wet tensile strength of paper improved by this treatment and tensile properties optimized at 10%SA, 5%SDHP and 1%ATC. Chemical treatment had an effect on cristallinity index and crystallite size of fibers. X-ray diffractometr demonstrated crystallinity index decreased compared to untreated pulp. FTIR analysis showed that carboxyl groups were slightly increased in treated fibers.

Key words: Succinic Acid · Wet tensile strength · Anionic trash catcher · Crystallinity index · XRD · FTIR

INTRODUCTION

A principal object of the present study is the provision of a method for adding wet strength to recycled type of paper. In earlier studies, wet strength resin was developed when wet strength resin was added directly to untreated fibers. However, for this study, treated fibers were used in order to study nothing. Fibre charge is an important factor that influences the papermaking process as well as the paper properties. Cellulose fibers contain various ionisable groups: carboxyl-, sulfonic acid-, phenolic- and hydroxyl groups and are therefore negatively charged at all pH values. Under normal papermaking conditions, the negative fibre surface or inside the cell wall; hence they are referred to as surface- and bulk charge, respectively. Surface charges are important for fibre- and paper strength [1]. It was shown that curing is necessary for reaction between the amino groups of PAE (wet strength resin) and the carboxyl groups of the fibres [2]. Several researchers have recently reported the use of specific oxidative treatments [3] and grafting treatments that increase fiber charge and fiber-fiber bonding [4, 5]. The tensile or sheet strength of fibrous products derived from cellulose fibers is due in large part to attractive fiber-fiber interactions. These interfiber interactions include hydrogen bonding interactions between fibers having hydrogen bonding sites. For cellulose, hydrogen bonding sites primarily include the hydroxyl groups of the individual cellulose chains. Treating cellulosic fibers with polycarboxylic acids is known in the art. For example, polycarboxylic acids have been used as crosslinking agents for cellulose. Cellulose has been modified by reaction with dicarboxylic acids and their derivatives to form simple diester crosslinks. Phtalic, Maleic and Succinic anhydrides have been used to form diester crosslinks in cellulose. Generally, the Catalyst promotes ester bond formation between the carboxylating agent and the cellulose fibers

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and is effective in increasing bond formation (the number of bonds formed) at a given cure temperature. Particularly preferred catalysts include alkali metal polyphosphates such as sodium hexametaphosphate and alkali metal hypophosphites such as sodium hypophosphite [6]. Also, Richard [6] showed that by treated fibers with Succinic Acid (6 percent by weight based on the total weight of fibers) and Sodium hypophosphite (3 percent by weight based on the total weight of fibers) and heated at the indicated temperature for 20 minutes, improved Water Retention Value (WRV) of fibrous composites. Sheet strength can be increased by enhancing the bonding of recycled furnishes with highly carboxylated fiber addition, by increasing cationic starch retention, or by increased retention of other cationic polymer dry and wet strength additives [7].

Succinic Acid (SA) changes the structure of fibers and these changes can increase carboxyl group, anionic charge and bonding capacity of fibers, therefore can improve strengthen paper specially wet strength of paper. Fourier transform infrared spectroscopy (FT-IR) and x-ray diffraction (XRD) are the methods used to study structural and chemical changes in fibers. FT-IR spectroscopy has been used a simple technique for analysis of wood samples and as a tool for monitoring structural wood changes during chemical or physical processing [8, 9, 10]. Stenstad *et al*. [11] modified the surface of the microfibrillated cellulose (bleached softwood sulphite pulp) by using different chemical treatment, such as GMA (Glycidil methacrylate). The modified microfibrillated cellulose was characterized with FTIR. The structure determination of cellulose by X-rays played an important role at the beginning of polymer science and the concept of long-chain molecules. The progress of structural research and the impact thereof can be evaluated and studied at certain stages by this development. Native cellulose of different sources, including wood, showed the same pattern, i.e., exhibited the same structure and it was also concluded that wood contains crystallites or microfibils of cellulose [12]. Infrared spectroscopy has been used to measure the amount of carboxymethyl cellulose (CMC) attached to cellulosic fibers. CMC was attached to an unbleached kraft pulp in aqueous [13].

The wet end disturbances have a chemical nature and they are usually related to dissolved and colloidal substances, which are called “DisCo’s”. The main DisCo’s in mechanical and recycled pulp suspensions are: hemicelluloses, pectins and wood resin (pitch). Today the water systems are quite closed and it leads to the accumulation of DisCo’s in the circulation waters. The dissolved and colloidal substances even contaminate the fibers and paper by sorption or deposition. It is very important to know the chemistry of the molecules and particles present in the wet end and how they interact together and with the added chemicals, fibres and fillers [14].

The present research studied the effect of Succinic Acid (SA) with two catalysts and two pH on the crystallinity index of cellulose and chemical properties of fibers. Crystallinity and chemical changes to fibers were assessed as a function of SA treatment and pH changes, using x-ray diffraction (XRD) and Fourier transform infra-red spectroscopy (FT-IR). Since papermaking process was occur on the natural pH, so in this research handsheets were made in natural pH.

MATERIALS AND METHODS

Materials

Pulp and Chemical: Recycled deinked Mixed Office Waste (MOW) pulp was prepared from *Latif* Paper Factory in Iran. Also, PAE which was produced by *BASF*, Germany and ATC which called *DICON FC* was produced by *GIG Chemie*, Germany, were obtained from this factory. Succinic Acid (SA) with a purity of 99.5% (*Merck*, Germany), Sodium dihydrogen Phosphate ($NAH_2PO_4.H_2O$) with a purity of 99% (*Merck*, Germany), Sodium hypophosphite or Sodium Phosphinate ($NAH_2PO_2.H_2O$) with a purity of 99% (*Merck*, Germany), Sulfuric acid (H_2SO_4) with purity of 97% (*Merck*, Germany) and sodium hydroxide with a purity of 98% (*Merck*, Germany) were supplied from Laboratory of Science and Research branch, Islamic Azad University, Tehran, Iran.

Methods

Treatment: In this research the experimental treatments were conducted based on earlier [6]. For treated fibers with Succinic Acid (SA) according to method of Richard [6], in the first stage prepared pulp was dewatered to about 30% consistency, after that and in the 1st experiment it was treated with 10% Succinic Acid and 5% Sodium dihydrogen Phosphate (SDHP) catalyst (half-one Succinic Acid), after 60 minutes, fibers heated at 140°C for 20 minutes. In the 2nd, 3rd and 4th experiment procedure of experiment one was repeated, but fibers treated by 6% Succinic Acid and 3% Sodium dihydrogen Phosphate catalyst, 10% Succinic Acid and 5% Sodium hypophosphite (SHP) catalyst, 6% Succinic Acid and 3% Sodium hypophosphite catalyst, respectively. Finally and before added ATC and PAE the pH of pulps was neutralized (6-8) with NAOH.

After treated fibers with carboxylating agent, fibers treated with cationic polymeric resin as ATC in two different batches 0 and 1%. Finally, after 10 minutes, 2% PAE was used as wet strength resin. All of handsheets plus control handsheet were treated with 2% PAE.

Treatment of fibers toward chemical analyses, after treated fibers Only with SA, the pH of fiber was naturalized by sodium hydroxide (NAOH). On the other hand, the pH of the fiber was changed to about 3.5 by Sulfuric acid (H_2SO_4). Then, chemical structure of fibers were investigated with FTIR and XRD analyses.

Measurement of pH: The pH of the samples was measured with a pH meter (*Metrohm*, Switzerland).

Handsheets: Handsheets were made for evaluation of wet tensile strength (by Lab Tech Semi-Automatic sheet machine) according to TAPPI Test Method 205 om-88.

Wet Tensile Strength Test: The wet tensile strength properties of hansheets were tested according with TAPPI Method T4560 om-03. This strength was performed by using *Lorentzen and wettern* (L&W), Sweden.

Chemical Analyses

XRD Analysis: The measurement of crystallinity in cellulose fibers was carried out by powder X-Ray Diffractometry (XRD, Philips xpert, Netherlands) as described by Segal *et al*. [15]. Intensities were recorded between 0° and 80° (2θ *-angle range*). Crystallinity (%) was defined as $(I_{002} - I_{am} / I_{002}) \times 100$, where I_{002} is the crystalline peak of the maximum intensity at 2θ between 22° and 23° for cellulose I and I_{am} is the minimum intensity at 2θ between 18° and 19° for cellulose I. The average size of crystallites was calculated by the Scherrer equation, Eq. 1. This is a method based on the width of the di?raction patterns in the X-ray refected crystalline region. In this study, the crystallites size were determined by using the difraction pattern obtained from 002 lattice planes of pulp samples,

$$D_{hkl} = k\lambda / (B_{hkl} \cos\theta) \quad (1)$$

where D_{hkl} is the size of crystallite, k is the Scherrer constant (0.84), λ is the X-ray wavelength and B_{hkl} is the FWHM (full width half maximum) of the refection hkl measured at 2θ, the corresponding Bragg angle [16].

FTIR Analysis: To determine the content of carboxyl groups in carboxylated fibers and carboxyl, methyl groups in carboxymethylated cellulose fibers, the dried powder of samples was analyzed by Fourier Transform Infrared (FTIR, Thermo Nicolet, USA) spectroscopy. A KBR disc technique was used. The range of wavenumber was calculated from 4000 to 400 cm^{-1}.

RESULTS AND DISCUSSION

FTIR Results: The IR spectra of pulp were obtained over a wavelength range of 400 to 4000 cm^{-1}. FTIR spectra of pulp treated with Succinic Acid (SA) and two catalysts with two pH (3.5 and natural pH) were compared with an untreated sample. Figure 1 through 5, show spectra of untreated pulp and pulp treated with SA. According to Heydarzadeh *et al*. [17], carboxyl, methyl and hydroxyl functional groups yield bands at wavelengths of 1618, 1426 and 1300 cm^{-1}. The absorbance peaks in the 3300- 3400 cm^{-1} region were attributed to the stretching of O-H groups, whereas those around 2800-2900 cm^{-1} were due to the stretching of C-H [18]. Carboxylation of cotton linters was investigated relative to its use in ion exchange and the effect of these treatments in cotton-linter fibers was considered in relation to the FTIR spectrum [19]. Stensted *et al*. [11] reported that for each GMA (Glycidil methacrylate) monomer added to the polymer chain a new ester group is introduced. The peak at 1722 cm^{-1} in FTIR spectrum shows that a large a mount of ester groups were introduced, indicated that polymer chains of GMA were grafted on microfibrillated cellulose. The peaks at 2916 and 2847 cm^{-1} were assigned to the asymmetric and symmetric C-H stretch modes of the $-CH_3$ and $-CH_2$ groups [20]. According to reported data, the peaks at wavelength around 1611 to 1642 cm^{-1} and 1730 to 1739 cm^{-1} was attributed to the presence of carboxyl group (-COO) and absorption band around 1323 to 1377 cm^{-1} and 3414 to 3587 cm^{-1} confirmed the presence of the hydroxyl group (-OH) [21]. In a study characterizing carboxymethyl cellulose and polyacrylamide graft copolymer, a peaks at 1603 cm^{-1} was attributed to the presence of carboxyl group and peaks at of 1325 and 1423 cm^{-1} were allocated to methyl and hydroxyl groups [22]. The peak occurs at 1740 cm^{-1} and can be assigned to the acid function and the peak occurs at 1434 cm^{-1} is in the fingerprint region and thereof cannot be assigned unambiguously [13]. The peak 1632 cm^{-1} in Blank sample showed the carboxyl group in fibers. The OH groups have a higher value in case of pulp treated with sodium hydroxide than untreated one. This is due to that, the sodium hydroxide decrease the hydrogen bonds between the OH groups of pulp. This can be confirmed by the shift of OH groups to the higher wave length [23].

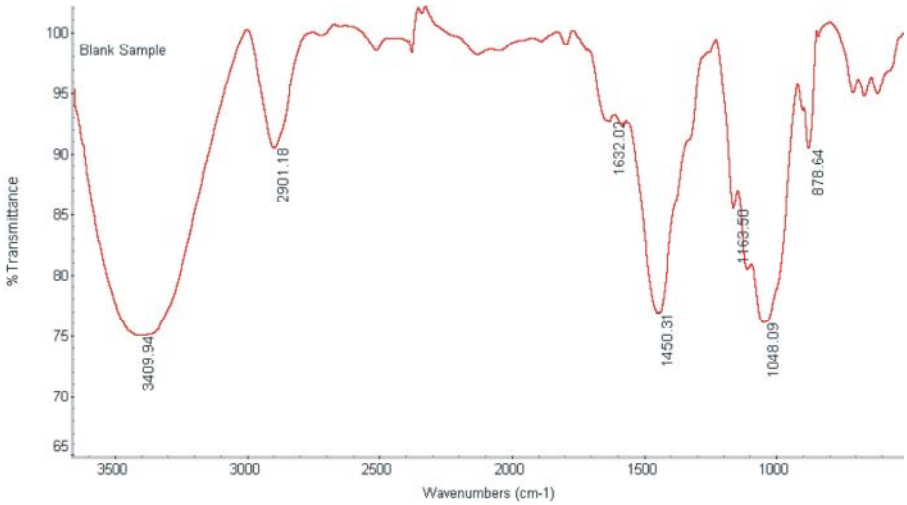

Fig. 1: FTIR of blank sample

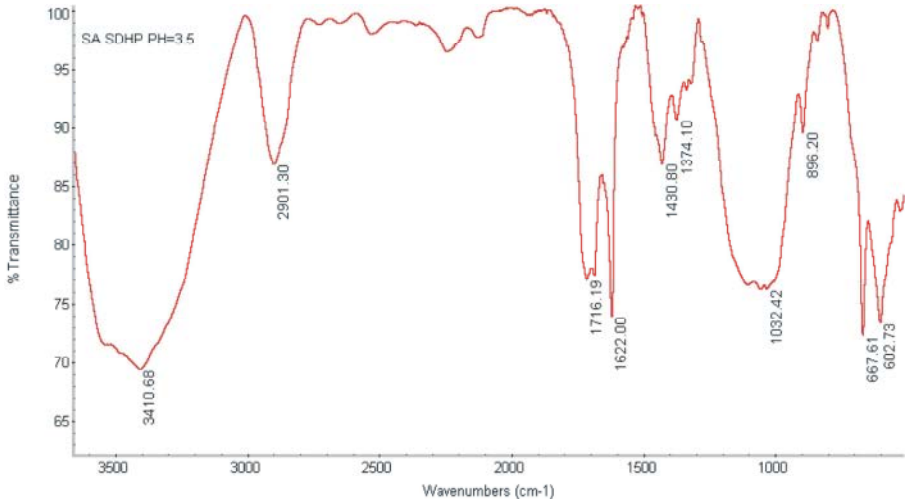

Fig. 2: FTIR of sample treated by SA with SDHP in pH=3.5

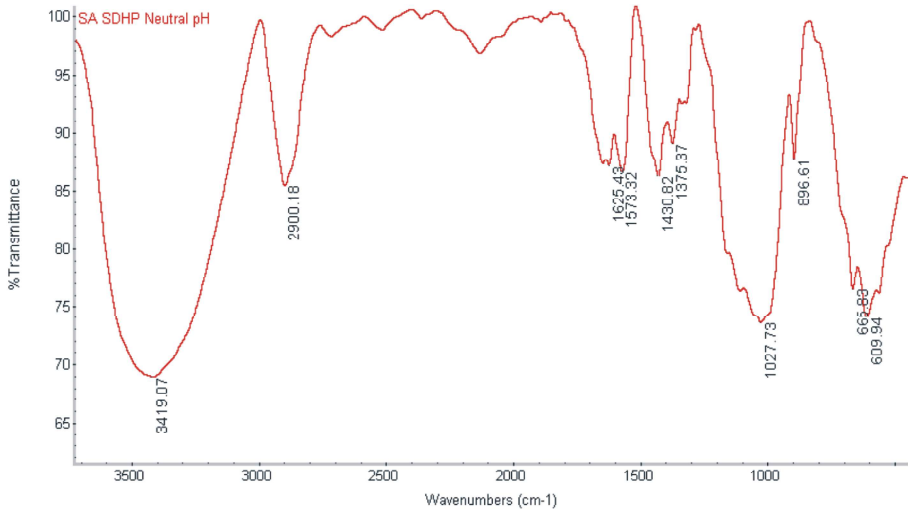

Fig. 3: FTIR of sample treated by SA with SDHP in pH=7

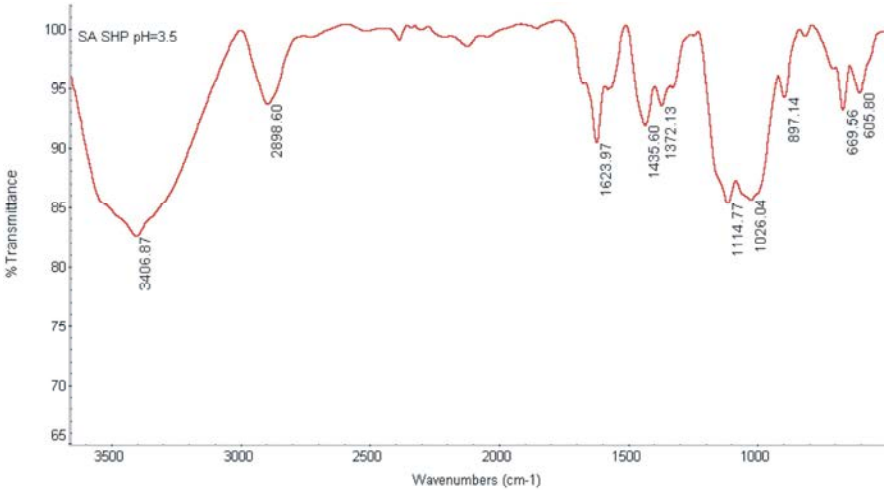

Fig. 4: FTIR of sample treated by SA with SHP in pH=3.5

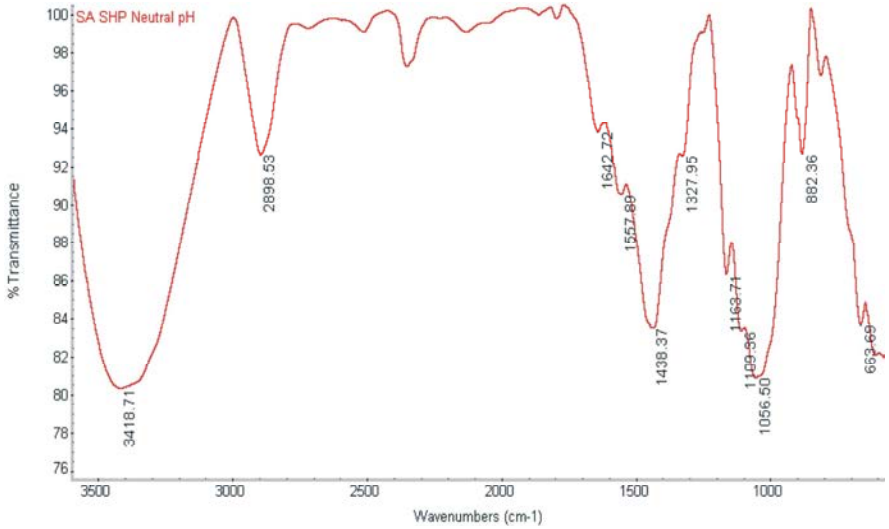

Fig. 5: FTIR of sample treated by SA with SHP in pH=7

Table 1: Infrared Spectra of Blank Sample and Treated (Carboxylated) Fibers

Material	Wavenumber (cm^{-1}) (-COO)	Wavenumber (cm^{-1}) (-OH)	
Blank sample	1632	-	3409
Sample treated by SA with SDHP in pH=3.5	1622		
	1716	1374	3410
Sample treated by SA with SDHP in pH=7	1625	1375	3419
Sample treated by SA with SHP in pH=3.5	1623	1372	3406
Sample treated by SA with SHP in pH=7	1642	1327	3418

Table 2: Relative Absorbance of Infrared Spectra from Treated (Carboxylated) Fibers

Material	Relative absorbance of COOH	Relative absorbance of OH
Blank sample	1.03	0.72
Sample treated by SA with SDHP in pH=3.5	0.82	0.74
Sample treated by SA with SDHP in pH=7	1.04	0.74
Sample treated by SA with SHP in pH=3.5	1.04	0.87
Sample treated by SA with SHP in pH=7	1.07	0.86

Table 3: Effect of carboxylation on the Crystallinity Index and Crystallinity Size of Cellulose in Treated and Untreated Fibers

Material	%Crystallinity index	Crystallite size (nm)
Blank sample	49	4
Sample treated by SA with SDHP in pH=3.5	47	4.7
Sample treated by SA with SDHP in pH=7	39	4.7
sample treated by SA with SHP in pH=3.5	27	3.8
sample treated by SA with SHP in pH=7	21	0.5

The treated samples (carboxylated) and untreated sample (blank sample) were analyzed by FTIR. As shown in Figs. 1 to 5, there were distinct increases in both the carboxyl groups in pulp samples treated with SA. Table (1) shows the different infrared data of untreated and carboxylated fibers. it is obvious that the broad absorption band around 3400 cm^{-1} is due to the stretching frequency of the hydroxyl group (-OH). From Table 3 it is clear that the crystallinity index of treated fibers was decreased. This can be confirmed by the shift that occurred for OH group at 3409 to 3418 and 3419 cm^{-1} in carboxylated fibers with neutral pH due to the decrease in hydrogen bonds by breakage of hydrogen bonds in the crystalline part of cellulose chains. Also, from the table, the crystallinity index of SA treated fibers without neutral pH was decreased. This may mean that the wave number of OH groups at 3409 cm^{-1} was decreased by increasing SA due to the decrease in the amorphous region. The bands around 1300 cm^{-1} are assigned to hydroxyl group (-OH) bending vibration. The presence of a strong absorption band around 1500 to 1700 cm^{-1} confirmed the presence of the carboxyl group (–COO). Table 2 show the relative absorbance (ratio of intensity of any band to intensity of band at 1325 cm^{-1}; Nada *et al*. [19] of COOH at 1720 cm^{-1} and OH 3439 cm^{-1}. This table show that treated fibers with SHP catalysts had higher relative absorbance of COOH and OH than fibers treated by SDHP catalysts. Also, this table show that neutral fibers had higher relative absorbance of COOH and OH. This is attributed to the increase of swelling of cellulose chains. This result showed the penetration of chemicals through cellulose chains and consequently increase the incorporation of carboxylic groups onto cellulose. The result of FTIR analysis showed that the fiber treated with SHP catalysts was better carboxylated during SA treatment than fibers treated with SDHP catalysts.

XRD Analysis: X-ray diffraction was used to investigation crystallinity of cellulose in samples. Table (3) shows the Crystallinity index and crystallinity size of Blank and treated samples (Figs. 6 to 10).

The result of XRD analysis showed that crystallinity was decreased in fibers after treated by SA. There was breakage of hydrogen bonds in the crystalline part of cellulose chains during carboxylated fibers. Crystallinity of cellulose treated with SDHP and in pH 3.5 was higher than fibers treated with SHP and in neutral pH this result might be attributed to the removal of extractive, lignin and other compound with amorphous nature or more degradation of hydrogen bond in the amorphous part of cellulose chains than the other ones. In samples without neutral pH (pH=3.5) treated fibers with acid due to the increase in the hydrogen bonds by degradation of amorphous parts in the cellulose fibers, according to Nada *et al*. [19]. Otherwise, NaOH that used for neutralization can cause cleavage of the hydrogen bonds in the crystalline part of cellulose chains and results to reduce the crystallinity according to Mohkami and Talaeipour [21]. This can be confirmed by the shift that occurred for OH group at 3409 cm^{-1} to high wave number of treated fibers in neutral pH (pH=7).

The crystallinity of cellulose fibers has been investigated in several studies. X-ray diffraction has been used for evaluation of crystallinity index in carboxylated cellulose and explain that treated fibers with acid cause an increase in the hydrogen bonds by degradation of hydrogen bond in the amorphous part of cellulose [19]. X-ray diffraction has been used for investigated of the percentage crystallinity in carboxymethylated and carboxylated fibers from waste paper [21]. According to Joonobi *et al*. [24] increase in the crystallinity index was expected after chemical treatment by removal of lignin and hemicelluloses, because of their amorphous nature. Kumar *et al*. [25] determined the crystalline content of nanoparticles increased from 37% (delignified raw materials) to 48% (nanoparticles). The crystallinity was found to increase owing to smaller particle size and removal of amorphous substances such as lignin, hemicelluloses and extractives.

Measurement Wet Tensile of Treated Fibers: According to Fig. 11, the treated handsheets with SA and two catalysts only and treated handsheets with a combination

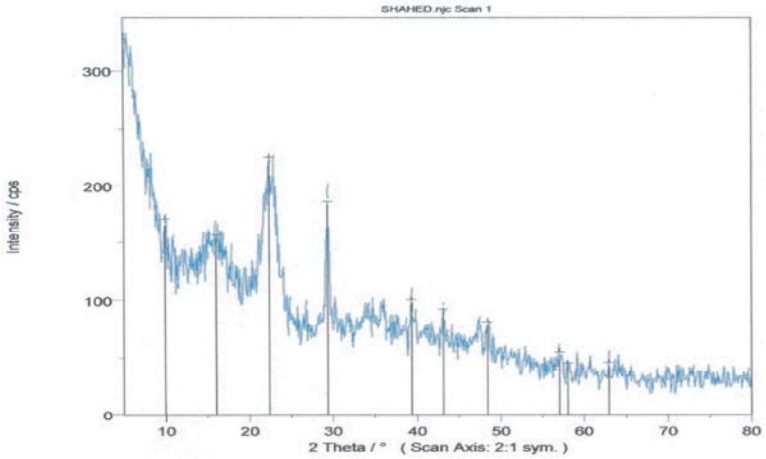

Fig. 6: XRD of blank sample

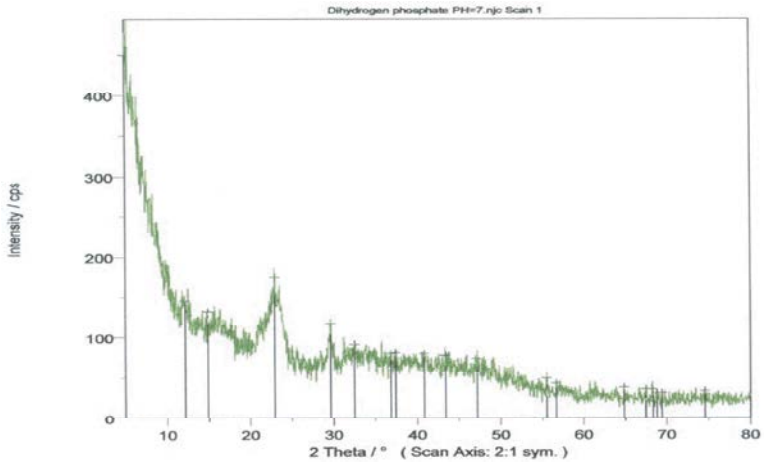

Fig. 7: XRD of sample treated by SA with SDHP in pH=3.5

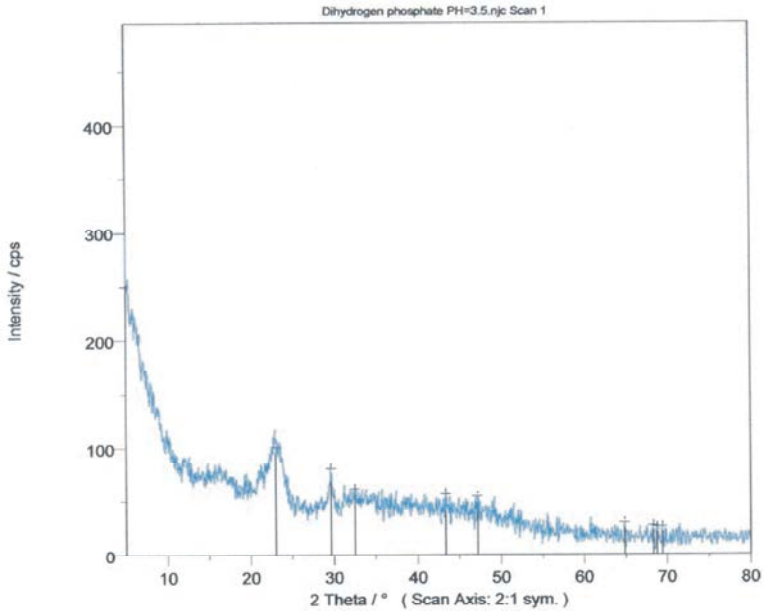

Fig. 8: XRD of sample treated by SA with SDHP in pH=7

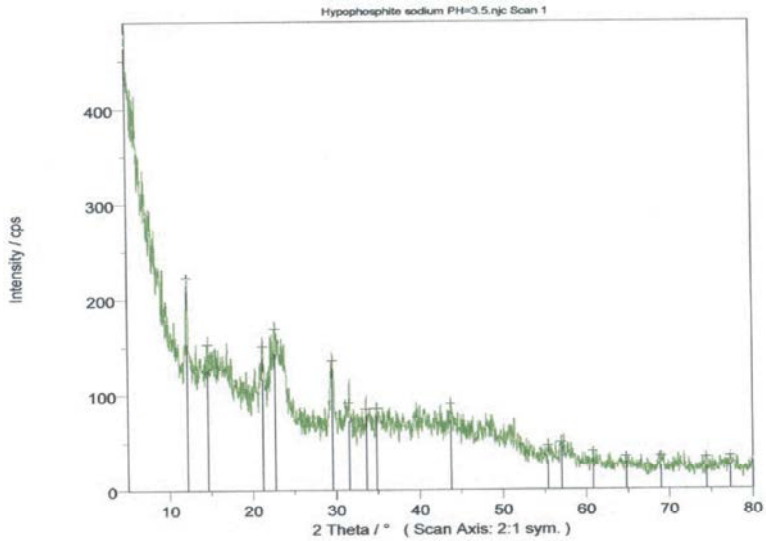

Fig. 9: XRD of sample treated by SA with SHP in pH=3.5

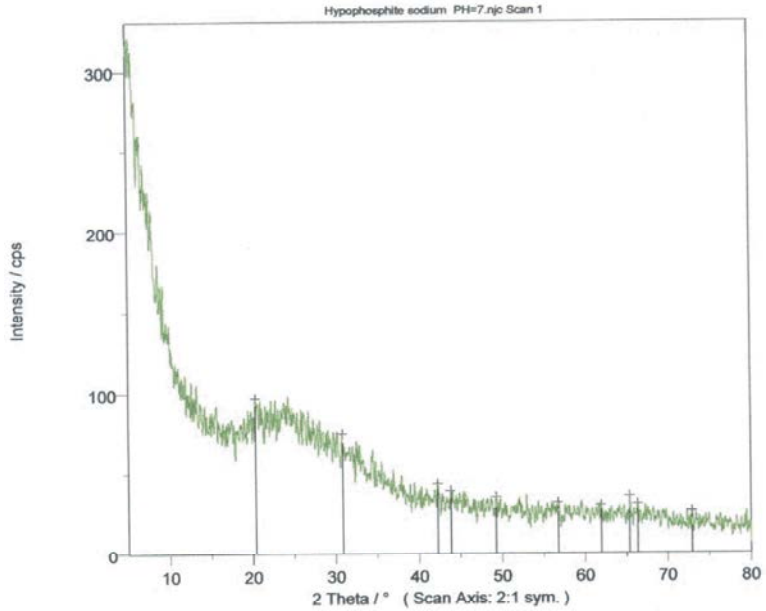

Fig. 10: XRD of sample treated by SA with SHP in pH=7

of SA and ATC showed higher wet tensile strength compared with control handsheet. It seems that applying SA in fibers leads to increase bonds and carboxyl groups into fibers, so the strength increased, especially at 10% SA (except handsheet that treated by 10% SA with SHP catalyst and 1% ATC). Also, Consumption of ATC cause to rise this strength dramatically in hadsheets made from treated SA and particularly with SDHP catalyst. Besides, based on the figure and according to investigations made for statistical results, it was observed that there was no meaningful difference between the wet strength of handsheet made from fibers that treated by 6% SA and SHP catalyst only. Regarding the higher wet tensile strength of treated hadsheets with combination of SA and ATC compared with control handsheet, it can be pointed out that SA treatment would cause to formation of ester bond and the catalyst promotes ester bond formation between it and the cellulose fibers. It was obtained that SDHP promotes ester bond formation better than SHP catalyst. On the other hand, treated fibers with ATC would cause the Dissolved and Colloidal Substances (DCS) that has anionic charge to be taken off and omitted

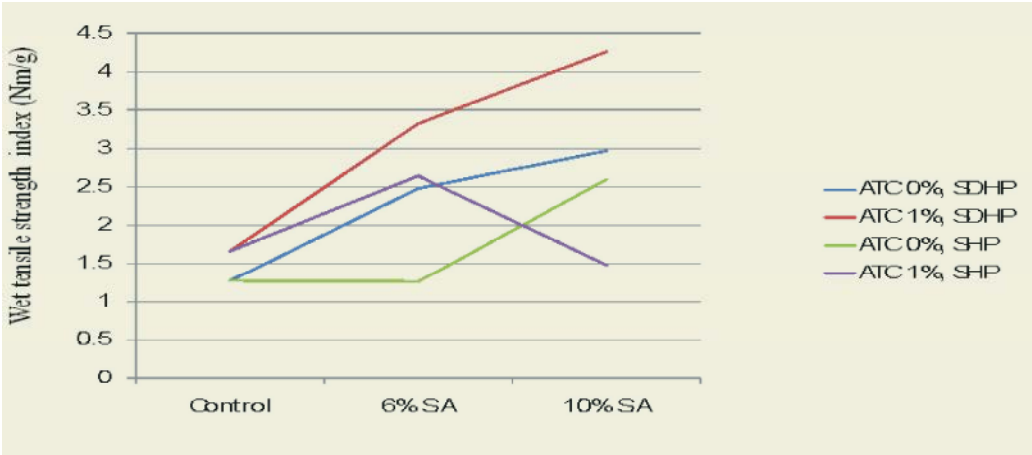

Fig. 11: Wet tensile strength index of treated hansheets by SA with 2 catalysts SDHP and SHP, and ATC

from the fibers, so, wet strength resin (cationic charge) can more effectiveness and improved the wet tensile strength of handsheets. As shown in Fig. 1, the highest strength of the handsheets was 4.26 Nm/g for the treated fibers with 10% SA and SDHP catalyst and 1% ATC. 1.27 and 1.66 Nm/g were wet tensile strength index for 0% and 1% ATC control handsheet, respectively. In contrast, the lowest strength of treated handsheets was 1.48 for the handsheet made from SA with SHP catalyst and 1% ATC treated fibers. It seems that for treated fibers ATC had positive effect on wet tensile strength of hansheets (except at 10% SA with SHP catalyst). The results from ATC treatment showed that wet tensile strength of handsheet dcreased at 10% SA with SHP catalyst compared with control handsheet treated with ATC, presumably due to reduce anionic sites of fibers, thus reduce the effectiveness of wet strength resin and decreased wet tensile strength of handsheet.

Fiber-to-Fiber interactions increased by SA treatments [6]. The tensile or sheet strength of fibrous products derived from cellulose fibers is due in large part to attractive fiber-to-fiber interaction [26]. The more charged groups that are attached to the fibres, the more they swell, thus increasing the flexibility and conformability of the fibres. An increased amount of xylan on the surface strengthens the paper, whereas kraft lignin and lipophilic extractives are known to impair the strength properties of paper [27]. Depending on the grafting agent employed, it was possible to increase the negative fiber charge by 50-100%, or alternatively, generate a fiber with a net positive charge. Physical testing of these biografted pulps demonstrated that these changes in fiber charge could yield distinct changes in physical strength properties including enhancements in burst, tensile, tear index and most notably a 30-60% increase in wet-strength values [5].

On the other hand, according to the Table 3 crystallinity index of treated fibers was decrease and it is confirmed by increase wet tensile strength in samples (Fig. 11). As seen in the figure 11 samples treated with SDHP catalyst has higher wet tensile strength compared with sample treated with SHP catalyst, but this unusual behavior could be brought about by other phenomena, such as the curling of the fibers and the deposition of solids onto the fibers. Sheikhi *et al*. [28] made this point: "The curling of the fibers and the deposition of solids onto the fibers caused to reduce the strength of paper, while the crystallinity was decreased". The decrease in the crystallinity index is attributed to the degradation of the cellulosic materials in the primary wall, whereas an increase in the tensile strength indicates the improvement in the packing order of the crystalline materials particularly in the S_1 layer. The tensile strength directly related to the crystallinity index [29].

CONCLUSIONS

- FTIR analysis indicated that functional groups of carboxyl had slightly increased. The presence of a strong absorption band around 1500 to 1700 cm^{-1} was confirmed the presence of carboxyl group (-COO).
- X-ray diffraction demonstrated that crystallinity index and crystallinity size of fibers were decreased during treatment. This is due to the cleavage of the hydrogen bonds in pulp during treatment.

- Results indicated that wet tensile strength of paper improved by SA treatment and highest wet tensile strength was shown in sample treated by 10%SA, 5%SDHP and 1%ATC.

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