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Cellulose Fibers Dissolution in Alkaline Solution

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Abstract

In this study, NaOH dissolution method was applied to dissolve cellulose fibers which extracted from date palm fronds (type Al-Zahdi) taken from Iraqi gardens. In this process, (NaOH)-solution is brought into contact with the cellulose fibers at low temperature. Experiments were conducted with different concentrations of NaOH (4%, 6%, 8% and12%) weight percent at two cooling bath temperatures (-15 ºC) and (-20ºC). Maximum cellulose dissolution was 23 wt% which obtained at 8 wt% concentration of NaOH and at cooling bath temperature of -20ºC. In order to enhance the cellulose fibers dissolution, the sample was pretreated with Fenton's reagent which consists of hydrogen peroxide (H_2O_2), oxalic acid ($C_2H_2O_4$) and ferrous sulfate ($FeSO_4$). This reagent reacts with cellulose fibers and produces free radicals which increase cellulose dissolution. In this work three variables were studied: cooling bath temperature (-15ºCand-20ºC), NaOH concentration (4%, 6%, 8% and12%) and time of Fenton's reagent treatment (1-48) hrs. The results showed that the best percent of cellulose dissolution was (42 wt %) which occurred at treatment time (24 hours), temperature (-20ºC) and NaOH concentration 8%. In another set of experiments urea was added to NaOH solution as a catalyst with proportion (6%NaOH+4% urea) at two temperatures -15 and -20 ºC. The results show that the solubility of cellulose increase to 62% for the sample which treated with Fenton's reagent and to 35% for the untreated sample, both values were obtained at -15ºC.

Keyword: *Dissolution of cellulose, cellulose fibers, Fenton's reaction.*

1. Introduction

Dissolution of cellulose is a kinetic process where the forces that maintain the crystalline structure are overcome and lead to the disintegration of the structure into molecules [1]. The forces include the hydrogen bonds within the chain (intramolecular) and between the neighboring chains (intermolecular), as well as the van der Waals forces and hydrophobic forces. The dissolution occurs when such a solvent is introduced to polymer which is able to disturb the mutual interaction of the polymer molecules [2].

Cellulose is a linear, semi-flexible polymer that is self-organized in crystalline and non-crystalline phases. Since cellulose cannot melt, dissolution is a major issue. Cellulose solutions are used for processing cellulose in the form of fibers, films, membranes or other not too bulky objects such as sponges or aerogels, or for performing chemical derivatisation. Since cellulose chains have no specific features, the cellulose must be dissolved as occur to any other flexible or semi-flexible polymer, and cellulose solutions should behave as normal polymer solutions [3].

The development of cellulose dissolution/regeneration strategies constitutes an increasingly active research field. These are fundamental ways of several production processes

and applications. A wide range of appropriate solvents for cellulose is already available. However, most solvent systems have significant limitations, and there is intensive activity in both industrial and academic research aimed at improving existing solvents and developing new ones. Cellulose solvents have very different nature which gives great challenges in understanding the delicate balance between different interactions [4].

Previously, several traditional solvent systems were introduced. One of the famous methods to dissolve cellulose was CS_2 method (viscose type), which was the oldest and complex procedure for producing rayon [5]. In this way, a prior chemical modification of the molecules was needed, leading to serious environmental pollution and poor human health. Consequently, due to these defects, non-derivative solvent systems are more acceptable were used, inclusive ammonium fluorides /dimethylsulfoxide (DMSO), N, N-Dimethylacetamide/ lithium chloride (DMAc/LiCl), and also N-methyl-morpholine-N-oxide (NMMO) [6]. These three solvents accumulate with a several structure and are thought to dissolve the cellulose without breaking any existing chemical bonds.

Another solvent is sodium hydroxide used to dissolve cellulose, recently the main focus was placed on the aqueous alkaline solvents due to consideration of being inexpensive, harmless and eco- friendly, so sodium hydroxide can be regarded as the most suitable solvent for cellulose dissolution [7]. The purpose of the current study is to dissolve cellulose which extracted from date palm fronds after treated it with Fenton's reagent and sodium hydroxide solution with and without urea under controlled conditions.

2. Experimental Work

In order to carry out dissolution process of cellulose fibers, several steps must be conducted. First, the quantitative analysis of date palm frond was done to identify the cellulose content in the fronds. Secondly, the sample was delignificated using modified organosolv to remove lignin from biomass (date palm fronds). In the third step the sample was bleached to remove most of the remaining lignin and hence increasing cellulose content to about 96%). The last step includes dissolving cellulose fibers using NaOH solution with and without urea.

2.1 Compositional Analysis of Data Palm Fronds

The samples of date palm fronds were collected in the summer season from Babylon farms from a kind of date palm named (Al-Zahdi) and stored in pre- sterilized bags in refrigerator freeze. The Samples then taken to the laboratory for test and before being treated, impurities should be removed by washing them with distilled water several times .After that, the samples were shredded using electric cutter and crushed with humor then sieved. Fragments with a particle size of (270-90) μm were selected to be used in the next steps [8]. Table (1) shows the composition of fronds (leaves and stalk).

Table 1,
The composition of date palm fronds (leaves and stalk) based on dry weight wt/wt

Sample	Carbohydrate	lignin	Ash	Extractive	Protein
Leaves	52	26	8	10	4
stalks	59	23	7	8	3

2.2 Delignification

Lab scale experiments were performed in two liter autoclave batch reactor. The date palm fronds (biomass) were milled to about 0.18mm. A suspension of biomass-water-organic solvent (ethanol) was made (typically, 200 ml solvent per 20 g biomass). The experimental parameters in the delignification process were: temperature, ethanol /water ratio wt/wt and digestion time. From previous study, the optimum conditions for delignification process were: temperature 185ºC, ethanol: water 50:50 and digestion time 80 min [9]. In order to reduce the digestion time, three types of catalyst were tested: H_2SO_4, NaOH and Ca $(OH)_2$. Among them, sodium hydroxide (0.025M) was found the best one which reduces the time from 80 min to 30 min. The aim of delignification, is to remove lignin and hemicellulose and obtain pure cellulose [10].

2.3 Bleaching

Bleaching is a chemical treatment performed to different types of wood pulp to remove pulp color so that it becomes whiter. The objective of

chemical bleaching of the pulp is basically to remove all of the remaining lignin. So the process is often referred to as delignification [11]. Chlorine is the basis for the most common bleaching processes**.** Three grams of dried sample was weighted and put into 500 ml flask, then 250ml of sodium chlorite solution (60 gm per liter sodium acetate and 60 gm per liter sodium chlorite) was added, the sample was then soaked for four days[12]. After that it was washed with deionized water, followed by extraction with NaOH (0.15 N) several times and then it was washed with hypochlorite and distilled water. Finally, the sample was dried. It was noted that the color was changed from dark brown to white (this means eliminating the lignin and the remaining was mainly cellulose as shown in figure (1).

Fig. 1. The sample after bleaching.

2.4 Fenton's Reagent

Fenton's reagent is a solution consists of hydrogen peroxide with ferrous iron as a catalyst that is used to oxidize contaminants or waste waters. It was developed in the 1890s by Henry John Horstman Fenton as an analytical reagent. The traditional Fenton reaction is the catalyzed decomposition of dilute hydrogen peroxide (H_2O_2) by iron (II) to form radicals of hydroxyl.

$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + \bullet OH + {}^{-}OH$

2.4.1 Fenton's Reagent Preparation

Fenton's reagent can be prepared by mixing: (80mM H_2O_2, 0.5mM $FeSO_4$, 10mM oxalic acid, 10mM sodium oxalate), Mixing between oxalic acid and sodium oxalate was according to the following volume ratio: (1:13) to reach pH= 4.6-5 [13].

2.5 Cellulose Dissolution Procedure

1. Solutions of sodium hydroxide were prepared with different concentrations (6%, 8%, 10% and 12%) weight percent. For example to prepare 6% wt, 6 g of NaOH is completed to 100 ml by adding 94g distilled water [14].
2. The sample which bleached before was treated with Fenton's reagent by taking 0.2 g of the sample (mainly cellulose) with 10 ml reagent. Then placed in a plastic container (30 ml volume) and stirred for 24 hrs using magnetic stirrer [15]. After that the sample was washed with deionized water and dried for 8 hours at 50^{o}C. Figure (2) shows the sample after treatment with Fenton's reagent.

Fig. 2. The sample after treatment with Fenton's reagent.

3. The treated sample was placed in a test tube and 10 ml of NaOH solution was added.
4. The sample was left for 1 hr, and then placed in a plastic container which contains ethyl glycol (38%) and put in freezer for 24 hrs.
5. The sample was removed from the freezer and left for at least one hour at room temperature to melt it.
6. Then the sample was centrifuged at 2500 rpm to separate the dissolved fibers from those which undissolved.
7. Undissolved fibers were washed with distilled water and weighted to calculate dissolution percent as in eqn. (1).

$$\text{Cellulose dissolution\%} = \frac{\text{weight of cellulose}_{\text{before dissolve}} - \text{weight of cellulose}_{\text{after dissolve}}}{\textit{weight of } \text{cellulose}_{\text{before dissolve}}} * 100 \quad \ldots(1)$$

8. The dissolved fibers were injected in 10 % dilute sulphuric acid to form rayon, as shown in figure (3).

Fig. 3. Synthesis of rayon (regenerated cellulose).

3. Results and Discussion

3.1 Cellulose Dissolution

Three parameters were studied in the dissolution process; sodium hydroxide concentration, path cooling temperature and Fenton's reaction time. The ranges of these parameters were (4 %-12%) weight percent, two temperatures (-15 and -20) ºC and (1-48) hours respectively. Two types of samples were used; sample was treated with reagent and a sample without treatment. Experiments were repeated four times and average values of cellulose dissolution were taken.

3.1.1 Effect of Fenton's Reaction Treatment

To study the effect of Fenton's reagent use on cellulose dissolution, several values of Fenton's reaction time were studied within the range (1-24) hr. Fenton's reaction produces radicals which break the hydrogen bonds for cellulose and make it ready to dissolve in NaOH solution. From fig. (4) it was concluded that cellulose dissolution increased with increasing time until time 24 hr, beyond which the solubility of cellulose fibers begin to decrease. This is because the structure of cellulose fibers distorted and leads to decrease dissolution of cellulose fibers. The optimum value of cellulose dissolution was 30 wt % which occurred at reaction time 24hr. Budtova and Navard (2016) studied the dissolution of cellulose fibers for cotton and found that the perfect Fenton's reaction time was 18 hr. to give maximum cellulose dissolution of 23%.

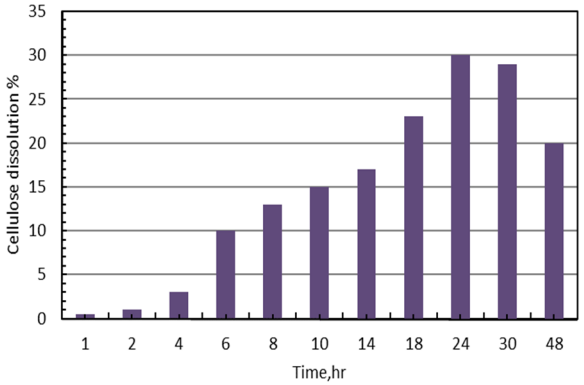

Fig. 4. Effect of Fenton's reagent duration on cellulose dissolution.

3.1.2 Effect of NaOH Concentration

To study the effect of alkaline concentration for cellulose dissolving, four values of concentration were taken (4%, 6 %, 8 % and 12 wt %). Two sets of experiments were conducted. In the first set, the samples were treated with Fenton's reagent for 24 hr. while in the second set the samples were untreated. Fig (5) shows that for both sets at (-15ºC) , the best value of NaOH concentration was 8% which causes cellulose dissolution of 30 wt% for samples which treated with Fenton's agent and 23% for samples which untreated. Wang (2008) was studied cellulose dissolution for cotton linter (as a cellulose fibers source) using NaOH solution with different concentrations and different cooling bath temperatures and found that the best cellulose dissolution percent was 20 wt% when using 9 wt% NaOH concentration.

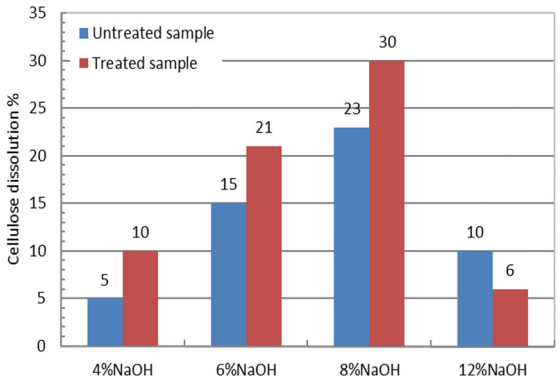

Fig. 5. Percentage of cellulose dissolution at different NaOH weight concentrations at temperature - 15 ±2°C.

The same two sets of experiments were conducted at -20°C. Figure(6) shows that the best cellulose dissolution for sample which treated with Fenton's reagent was 42% which occurred also at 8% NaOH concentration while for untreated sample the maximum cellulose fibers dissolution was 28% this implies that decreasing the temperature to -20°C will enhance the solubility of cellulose in NaOH solution.

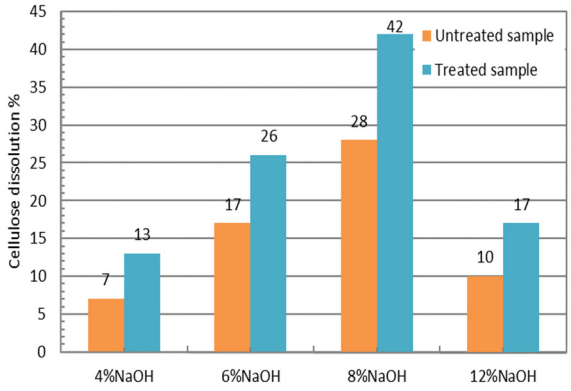

Fig. 6. Percentage of cellulose dissolution at different NaOH weight concentrations at temperature -20 ±2°C.

From figures (5and 6), it was concluded that the best NaOH concentration for cellulose dissolution process was 8%. Furthermore, the samples which treated with Fenton's reagent show better dissolution in NaOH solution than those which untreated.

Figure 7 illustrates how the hydrodynamic of hydrate ions was affected by the concentration of ion solution. At low solution concentration, the ions are surrounded by a large amount of water molecules and thus pairs of separated ions are existing. At high solution concentration, the ions will be surrounded by fewer amounts of water molecules resulting in lower hydrodynamic diameter. The cellulose crystallite diameter is 10 nm and inter-sheet distance is of about10 Å [17]. At low concentration of NaOH, the hydrodynamic diameter is too large to penetrate cellulose crystalline region, while at high NaOH concentration the hydration of NaOH will be insufficient to break hydrogen bonding which makes the cellulose to mercerize (not dissolved). Therefore; there is a certain concentration of NaOH solution which is appropriate to penetrate the cellulose and start to dissolution. This value of concentration is called turn point concentration (critical concentration) [18]. In the current study, it was found that the turn point concentration was 8%. This finding is agreement with that found by Wang (2008) who found that the turn point concentration of cellulose (cotton linter) was 9%.

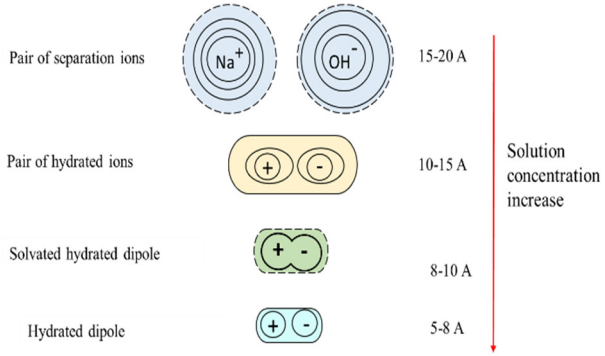

Fig. 7. Hydrodynamic diameter of hydrates with respect to NaOH solution concentration [19].

3.1.3 Effect of Temperature

With a view to study the effect of temperature on solubility of cellulose fiber, two values of temperatures were used (-15 °C and -20 °C) depending on previous studies [20] [21]. Figure (8) shows that at -20 °C, the best cellulose dissolution for sample which treated with Fenton's reagent was 42% and for untreated sample the maximum cellulose fibers dissolution was 28%. While at temperature (-15°C), the cellulose dissolution percent was 30 % for treated sample and 23% for untreated sample. This implies that decreasing temperature to (-20 °C) will enhance the solubility of the cellulose in NaOH solution.

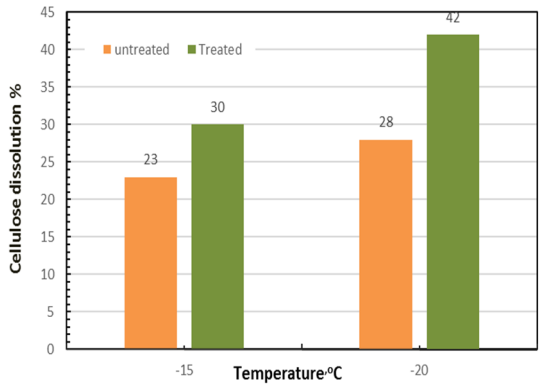

Fig. 8. Effect of temperature on cellulose dissolution at 8% alkaline solution.

The explanation the effect of temperature on the solubility of cellulose will be in the next section.

3.1.4 Effect of Urea Addition

The effect of adding urea to NaOH solution on cellulose fibers dissolution was studied, a solution of NaOH and urea with proportion (6%NaOH and 4% urea) was used to enhance cellulose fibers dissolution or stimulate crystalline polymerization [17].Figure (9) shows that at temperature -15°C the maximum cellulose fibers dissolution was 62% for the samples which treated with Fenton's reagent and 35% for untreated. Figure (10) shows the results of cellulose dissolution at temperature -20°C. For the sample which treated with Fenton's reagent the maximum cellulose fibers dissolution was 35% while for an untreated sample was 28%. These results for treated samples indicate that adding urea will increase the solubility from 30% to 62% at (-15°C) while at (-20°C) adding urea will decrease the cellulose fibers dissolution from 42% to 35%.

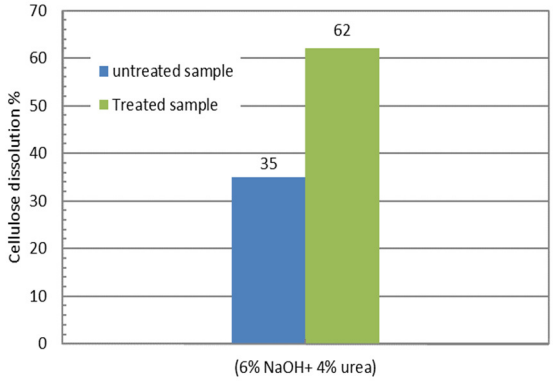

Fig. 9. Effect of urea addition on cellulose dissolution at temperature - 15 ±2°C.

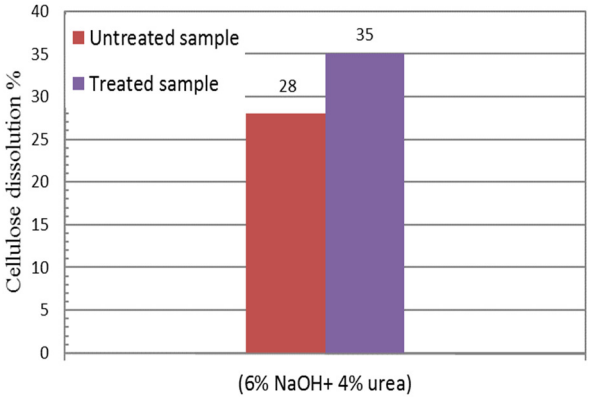

Fig. 10. Effect of urea addition on cellulose dissolution at temperature -20 ±2°C.

The cellulose fibers dissolution is a thermodynamic process and can be represented by Gibbs free energy equation:

$$\Delta G = \Delta H - T.\ \Delta S \quad \ldots (1)$$

For cellulose dissolution process (breaking cellulose crystal) by NaOH solution without urea, values of entropy and enthalpy change are -2.23 kJ /mol K and -495.48 kJ /mol respectively. While for the treatment with NaOH and urea, values are -0.546 kJ /mol K and -61.83 kJ /mol respectively [22]. At temperature 258 k (-15°C), ΔG=80.55 kJ /mol for treatment without urea and ΔG=79.01 kJ /mol for treatment with urea. The decrystallization reaction is exothermic because of the negative value of enthalpy so lower temperature is favorable. Figure 11 shows the plot of Gibbs free energy verses temperature for dissolution of cellulose by NaOH solution with and without urea. The two straight lines intersect at temperature 258k (-15°C). At temperature greater than 258K (-15oC), the treatment with NaOH and urea solution has lower ΔG than that without urea which is better for decrystallization reaction. While at temperatures less than 258k (-15°C) NaOH without urea has lower ΔG so it is better for decrystallization. In the current study the lowest temperature was 253K (-20°C).

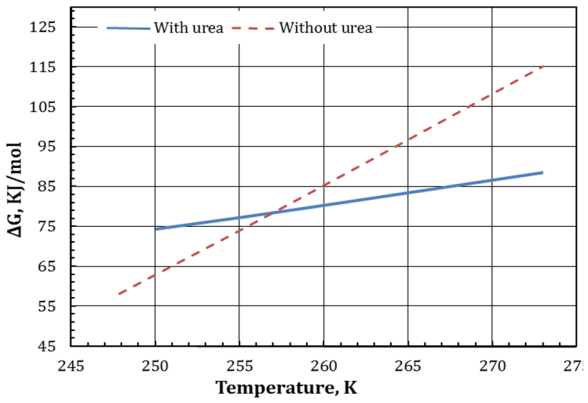

Fig. 11. Gibbs free energy for cellulose dissolution by NaOH with and without urea [16].

4. Conclusion

Based upon the results obtained so far, there is a possibility to dissolve the cellulose which extracted from the date palm fronds using NaOH solution method. The more effective parameters in the cellulose fibers dissolution process were: cooling bath temperature, NaOH concentration and time of Fenton's reagent treatment. The best NaOH concentration for cellulose fibers dissolution was 8 wt% at -20°C at which the dissolution of cellulose reaches 42% when the samples were treated with Fenton's reagent for 24 hr, while for samples untreated with Fenton's reagent the dissolution reaches 28%. Adding urea to NaOH solution (6%NaOH and 4% urea) was found to increase the cellulose dissolution to 62% at temperature -15°C for samples which treated with Fenton's reagent for 24 hr.

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أذابة ألياف السيليلوز في المحاليل القاعدية

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الخلاصة

في هذه الدراسة، تم أستخدام محلول هيدروكسيد الصوديوم لأذابة الألياف السليلوزية المستخلصة من سعف النخيل (نوع الزهدي) المأخوذ من المزارع العراقية. في عملية الاذابة، يتم تفاعل محلول هيدروكسيد الصوديوم مع ألياف السليلوز في درجات حرارة منخفضة. أجريت التجربة مع تراكيز وزنية مختلفة من هيدروكسيد الصوديوم (٤٪، ٦٪، ٨٪ و ١٢٪) في درجتين حرارية - ١٥ درجة مئوية و- ٢٠ درجة مئوية. كان الحد الأقصى لذوبانية السليلوز هي ٢٣ ٪ نسبة وزنية وقد تم الحصول عليها عند تركيز ٨٪ من هيدروكسيد الصوديوم وفي درجة حرارة -٢٠درجة مئوية. من أجل تعزيزذوبانية ألياف السليلوز ، تم معالجة النموذج مع كاشف فينتون الذي يتكون من بيروكسيد الهيدروجين (H_2O_2) وحامض الأوكساليك ($C_2H_2O_4$) وكبريتات الحديد الثنائية ($FeSO_4$). يتفاعل هذا الكاشف مع ألياف السليلوز لينتج الجذور الحرة التي تزيد من ذوبان السليلوز. في هذه الحالة تم دراسة ثلاثة متغيرات وهي درجة الحرارة عند (-١٥ و -٢٠) درجة مئوية، تركيز هيدروكسيد الصوديوم (٤٪، ٦٪، ٨٪ و ١٢٪) ووقت المعالجة بكاشف فينتون (٤٨-١) ساعة. وأظهرت النتائج أن أفضل نسبة لذوبانية السليلوزهي (٤٢٪) عند زمن معالجة (٢٤ ساعة) ، درجة حرارة (-٢٠درجة مئوية) وتركيز هيدروكسيد الصوديوم ٨٪. في مجموعة أخرى من التجارب تمت إضافة اليوريا إلى محلول هيدروكسيد الصوديوم بوصفه محفزا بنسبة (٦٪ هيدروكسيد الصوديوم + ٤٪ اليوريا) عند درجتين حرارية هي (-١٥ و -٢٠) درجة مئوية. أظهرت النتائج أن ذوبانية السليلوز تزداد إلى ٦٢٪ للنموذج الذي تم معاملته مع كاشف فنتون و٣٥٪ للنموذج غير المعالج، وكلتا القيمتين لذوبانية السيليلوز تم الحصول عليها عند درجة حرارة -١٥درجة مئوية.