

## Effect Of Extraction Conditions On The Yield Of Hemicellulose And Cellulose From Sunflower Stalks

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### Abstract

Various conditions for the extraction of hemicellulose and cellulose from sunflower stalks were examined, including delignification time concentration and time of alkaline extraction and ratio of liquid to dry sample. A procedure using one hour delignification at 70°C followed by extraction with 5% NaOH at 25°C for six hours gave the highest yield of cellulose and hemicellulose. The monosaccharide composition of the hemicellulose was determined by means of gas chromatographic analysis. Xylose was the predominant sugar of hemicellulose. Arabinose, rhamnose, mannose, galactose, and glucose were also present as minor constituents.

### Introduction

The major polysaccharides in higher land plants are cellulose and hemicellulose. Cellulose is the most abundant organic raw material available in the world today and it is naturally regenerable. Cellulose, its by-products and derivatives have always held an important place scientifically and commercially. Their value can only increase as other raw material

become depleted(1). The stalks of agricultural plants are a readily available source of both cellulose and hemicellulose. Sunflower stalk contains small amount of extractives and crude protein but large amounts of cellulose, hemicellulose and lignin (2,3,4).

The method of cooking such as soda pulping (alkaline), Sulfite pulping (acide) or semichemical pulping (NSSC) of the raw material greatly influences the properties of the pulp and paper produced from it(5). The alkaline degradation of polysaccharides during alkaline pulping is an important reaction which leads to extensive losses of polysaccharide material(6). Also, small amount of the polysaccharide material lost during the delignification treatment has been reported(7).

The present studies investigate the losses in the cellulose and hemicellulose materials from the sunflower stalk that are solubilized and normally lost during the delignification and alkaline treatments. This work is a part of a project to using the sunflower stalks as a cellulosic source in pulp and paper industry.

### Materials And Methods

Dry stalks of the sunflower were ground to flour using arestschMuhle mill (F.G. Bode and Co. W. Germany) fitted with a 20 mesh sieve. Samples of (25 gm) were delignified with sodium chlorite and acetic acid (8) to different periods (1.2 and 3h) at 70C°. The delignified samples were treated for 6 hours with various concentration of sodium hydroxide (2.5, 5.0, 10.0, 15.0, and 20.0% ) at 25C° under nitrogen. Then, the delignified samples were subjected to 5% sodium hydroxide at reaction times (1.5, 3.0, 6.0, 12.0, 24.0 and 48.0 hours). And finally the ratio of sample to alkaline liquid (1:10, 1:20 and 1:40) were examined.

Gas chromatographic analysis of carbohydrates has been performed by a number of methods. The most widely used being that involving trimethylsilyl ether. Whose major advantage is their rapid formation. Nevertheless, Water reacts with the silylating reagents and hydrolysis the silylated products, and thus the sample to be derivatized needs to be reasonably dry. Moreover, each individual sugar gives at least two peaks because of the various anomeric forms of each sugar in solution (9). As alternatives to the aforementioned derivative, the alditol acetates (10) and the aldonoitrile acetates (11) are widely used. Each sugar derivative give only one peak and has good chromatographic properties. The hemicellulose yield from each treatment was treated with

0.5M H<sub>2</sub>SO<sub>4</sub> at 100C° for six hours prior to conversion to alditol acetate derivatives before being examined by gas chromatography (G.C.) (12). Samples of cellulose were digested with 72% H<sub>2</sub>SO<sub>4</sub> for 1.5 hour at 20C° and following dilution to molarity. Were then heated at 100C° for 2.5 hours (13). The sugars were determined as their alditol acetates and examined by G.C.

### Results and Discussion

The flour of sunflower stalks were delignified by treating with hot aqueous acetic acid and sodium which generate chlorine and chlorine dioxide. Such treatment was carried out to oxidize and to dissolve the more soluble degraded lignin. Table 1 shows the cellulose and hemicellulose contents of sunflower stalks at different times of delignification. The results indicate a three hours treatment with sodium chlorite was sufficient to cause considerable degradation of hemicellulose while. Short time (one hour) delignification was sufficient to remove lignin in sunflower stalk. The final product (residue) after delignification treatment was a nearly white fibrous material in each treatment, while, a brown material was recovered by using rejected due to incomplete removal of lignin from the raw material. On increasing the time of delignification treatment, the losses in the hemicellulose content increase. Several investigators (14,5) stated that partial degradation of hemicellulose may have taken place during delignification.

During alkaine treatment the hemicellulose released at rates that depend on the concentration of the alkaline . for quantitative comparisons, a balance has to be struck between the conditions leading to complete release, and those giving acceptable low degradation of the hemicellulose materials. To examine the effect of alkaline concentration on the yield of cellulose and hemicellulose. Five concentrations of NaOH were used in the extraction procedure. (Table 2) shows the yields of cellulose and hemicellulose resulted for each treatment. The solubility of hemicelluloses reached a maximum at an alkaline concentration of 5% and further increases in concentration did not improve the extraction yield. Therefore, a 5% concentration of NaOH was used for extraction of hemicellulose from sunflower stalk in subsequent experiments.

The proportions of cellulose and hemicellulose released after different periods of alkaline treatment of the delignified holocellulose from the sunflower stalks are shown in table 3. The optimum period of extraction with 5% NaOH at 25°C° was six hours. Before then clearly not all of the hemicellulose had been released, and by then there had been partial degradation of the hemicellulose content. Alkaline extraction of hemicellulose was carried out at 25°C°. The hemicellulose has alimited solubility in cold alkaline solution, but warm alkaline solution lead to hemicellulose degradation, conse-

quently, alkaline extraction at room temperature was used to isolate hemicellulose for physico-chemical characterization (15).

The effect of sample to alkaline ratio on the yield of cellulose and hemicellulose was examined. Three different ratios were used (1:10 1:20 and 1:40 solid to liquid, respectively). Similar and optimum yield was found in the second and third ratio (Table 4). The large value of liquid was needed because the lightness of sunflower stalk and a great water absorption of pith.

Quantitative estimation were made of the natural sugar composition of cellulose and hemicellulose in each extract. The monosaccharide content of hemicellulose material were determined to G.C. analysis after hydrolysis and conversion to alditol acetates (Tabel 5). All samples contained similar monosaccharides compositions of hemicellulose. The predominant sugar of hemicellulose was xylose, although arabiniose, rhamnose, mannose, galactose and glucose were also present. Glucose was the only monmsaccharide found in the cellulose extract from all treatments. The result indicate that the cellulose is pure. The present investigation of various conditions for extracting and isolating cellulose and hemicellulose from sunflower stalks was revealed that the one hour delignification at 70°C° and 5% NaOH for extraction of hemicellulouse at 25°C° for six hours was the optimum condition.

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تأثير ظروف الاستخلاص على حصىلة الهميسلوز والسلوز الناتج من سيقان زهرة الشمس  
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تلتها معاملة ٥٪ هيدروكسيد الصوديوم على حرارة ٢٥٠م° ولدة ٦ ساعات. وتم الكشف عن مكونات الهميسلوز من السكريات الاحادية باستخدام تقنية الكروتوكراف الغازي. وقد اتضح بان الزايلوز هو السكر الرئيسي المكون للهميسلوز وكذلك وجد الارابينوز والرامينوز والمائوز والكالاكتوز والكلوكوز كسكريات احادية موجودة بنسب قليلة في الهميسلوز المستخلص.

#### الخلاصة

تم اختبار ظروف الاستخلاص المختلفة للهميسلوز المنتج من سيقان زهرة الشمس والتي تشمل فترة ازالة اللكتين وتركيز القاعدة والزمن ونسبة سائل الاستخلاص الى وزن النموذج الجاف. لقد بينت النتائج ان اعلى حصىلة للهميسلوز والسلوز كانت عند المعاملة لمدة ساعة واحدة لازالة اللكتين على حرارة ٧٠م° والتي

Tabel 1. Effect of delignification periods on the percentage of cellulose and hemicellulose obtained from the sunflower stalks.

Cook No.	Delignification time (hour)	Cellulose (%)	Hemicellulose (%)
1	1	40.20	30.72
2	2	40.35	30.88
3	3	39.72	24.72

Table 2. Effect of sodium hydroxide concentration on the percentage of cellulose and hemicellulose obtained from the sunflower stalks

Sodium hydroxide (%)	Cellulose (%)	Hemicellulose (%)
2.50	40.48	28.20
5.00	40.40	30.70
10.00	40.10	28.70
15.00	38.00	26.20
20.00	37.84	23.80

Tabel 3. Percentage of cellulose and hemisellulose obtained from sunflower stalks with 5% NaOH for different periods

Reaction time (hour)	cellulose (%)	Hemicellulose (%)
1.50	39.84	25.50
3.00	40.16	27.40
6.00	41.44	30.44
12.00	40.20	28.72
24.00	38.68	22.84
48.00	36.30	20.70

Table 4. Effect of solid to liquid on the recovery of cellulose and hemicellulose from sunflower stalks using 5% NaOH for six hours

Ratio of solid to liquid	Cellulose (%)	Hemicellulose (%)
1:10	39.08	27.60
1:20	40.72	30.50
1:40	40.92	30.10

Tabel 5. Monosaccharide composition of hemicellulose of sunflower stalks

Sugar	Percentage
Xylose	90.2
Arabinose	3.2
Rhamnose	2.1
Mannose	2.2
Galactos	1.2
Glucose	1.1