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COMPARATIVE PULPING OF SUNFLOWER STALKS

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The procedure of holocellulose content determination in non-wood plant raw materials was developed. The strength properties of pulp obtained from sunflower stalks by neutral-sulphite, soda, alkaline sulphite-anthraquinone-ethanol and peracetic methods of delignification were studied. Methodology of comparison of plant materials delignification methods using new lignin-carbohydrate diagram was proposed. It was shown, that the alkaline sulphite-anthraquinone-ethanol method of pulping is characterized by the highest delignification degree and is the most efficient among the studied methods

Keywords: sunflower stalk, holocellulose, delignification, lignin-carbohydrate diagram, delignification degree

Розроблено методику визначення вмісту холоцелюлози в недеревних рослинній сировині. Вивчено показники якості целюлози, отриманої з стебел соняшнику нейтральним-сульфітним, натронним, лужно-сульфітно-спиртовим та пероцтовим способами делігніфікації. Запропонована методика порівняння різних методів делігніфікації рослинної сировини з використанням нової лігнін-вуглеводної діаграми. Показано, що лужно-сульфітно-спиртовий спосіб варіння целюлози характеризується високим ступенем делігніфікації і є найбільш ефективним серед вивчених методів

Ключові слова: стебла соняшнику, холоцелюлоза, делігніфікація, лігнін-вуглеводна діаграма, ступінь делігніфікації

1. Introduction

Living standards improvement requires increase in quality and quantity of consumer goods, in particular paper and paperboard. To produce them it is necessary to increase the volumes of primary pulps. For the countries with limited free timber resources non-wood plant raw materials are used in order to increase pulp and paper production [1–3]. Non-wood plant raw materials also can be applied as an effective alternative to constantly decreasing forest resources in different regions. In countries with developed agriculture millions tones of by-products suitable for obtaining pulp are produced by cereal and technical crops treatment. Potential world resources of non-wood plant raw materials exceed 2.5 billion tons per year [4] and renew annually. Using non-wood plant raw materials in pulp and paper industry is beneficial in terms of environmental and socio-economic aspects. Wheat [5], rice [6] and canola [7] straw, abaca [8], bagasse [9], corn and reed [10], cotton [11], hemp [12], kenaf [13], miscanthus [14], elephant and switch grass [15], rapeseed straw [16], etc. have become widely spread as the types of non-wood plant raw materials for obtaining pulp.

2. Analysis of published data and problem definition

Sunflower is the most popular plant in many countries and is one of the profitable technical crops with high level of cost-effectiveness among agriculture crops. Sunflower is the third most cultivated oil plant in the world [17]. According to the US Department of Agriculture its production will take to 39.8 million ton during the periods of 2013–2014 year, which is relative increase of 9 %, compared to last year [18]. Such increase results from the more efficient cropping techniques and increase in overall sunflower plantings.

In worldwide practice of pulp and paper industry the alkaline methods, in particular soda, sulfate and neutral-sulphite processes have become the most common

cooking methods of obtaining pulp from lignocellulose materials [19]. Different methods of more ecological organosolv pulping are widely developed and applied [20–22]. Each of these methods has its advantages and disadvantages by physical and mechanical, ecological and economic indices.

Comparison of methods for pulping non-wood plant raw materials, including sunflower stalks, is devoted to number of works [10, 23, 24] with analysis of yield, kappa number, viscosity, bonding abilities of fibers, physical and mechanical indices of resulting pulps, its characteristics of beating and bleaching, etc. However, lignin-carbohydrate diagrams as well as comparing lignin extraction indices from plant material for additional evaluation of different delignification methods efficiency can be used.

3. The aim of the study

Therefore, the aim of this study is to obtain and compare yield, residual lignin content, strength properties of resulting pulps, to determinate delignification degree, construction and analysis of new lignin-carbohydrate diagram for different sunflower stalks delignification methods to determine a more efficient one.

4. Experimental Sections Materials and methods

4. 1. Raw materials

Sunflower (*Helianthus annuus*) stalks of 2012 harvest were taken in Sumy region of Ukraine in late autumn and were used for obtaining pulp. The stalks were cut at 0.1 m from the ground level and the leaves were trimmed. For the research purpose the stalks were cleaned out of leaves, cut at height of 20±5 mm and stored in desiccators to maintain constant humidity and chemical composition. Pith was eliminated because it contributes to various problems during cooking such as requirement of additional consumption of chemicals, increasing the foam, deterioration in the

quality of pulp and paper [25] and problems related to drainage [26].

Stalks of corn, wheat straw and rapeseed were used in order to perform comparative analysis of chemical composition. Corn (*Zea mays*) is one-year cereal growing in all regions of the world and has a gross rounded stalk up to 6 m, thickness 2–7 cm with core, which is filled of parenchyma cells (pith). In this work, the corn stalks from the Cherkassy region of Ukraine were used. For the research purpose the stalks were cleaned out of leaves, cut at height of 20 ± 5 mm, pith was eliminated and prepared materials stored in desiccators to maintain constant humidity and chemical composition.

Wheat straw (*Triticum vulgare*) is an annual grass of the cereal family (*Gramineae*, *Poaceae*) and is widely used not only for fuel production, but also as feed, livestock bedding, fodder and takes leading position in the processing capacity among non-wood raw materials in pulp and paper industry. Rapeseed (*Brassica napus*) belongs to herbaceous plants of the family (*Brassicaceae*). It is extremely important oil plant used mainly for obtaining of biodiesel. For the research the stalks of wheat straw and rapeseed from Vinnitsa region of Ukraine were cleaned out of leaves, cut at height of 20 ± 5 mm and stored in desiccators to maintain constant humidity and chemical composition. The moisture content of all lignocelluloses' materials was $7\pm 1\%$.

4. 2. Chemical analysis

Stalks of sunflower, corn, wheat straw and rapeseed were grinded in Wiley mill to produce sawdust on 40/60 mesh, which was analyzed for chemical composition. It was identified according to existing TAPPI standard procedures for different components [27], namely: T 211 om-07 for ash, T 428 om-08 for hot-water extractives, T 212 for 1 % NaOH soluble substances, T 204 cm-07 for alcohol-benzene extractives, T 222 cm-01 for pentosanes, T 222 om-02 for Klason lignin and cellulose by the Kürschner-Hoffer method [28]. All chemical analyses were carried out twice allowing to calculate the mean values and standard deviations, which do not exceed 5 %.

Holocellulose content was identified by TAPPI T 203 cm-09 standard procedure with our modification accounting peculiarities of anatomical structure and chemical composition of annual plants. The key point of the modification was 30 minutes treatment of preliminary extracted by alcohol-benzene non-wood samples by 10 % peracetic acid at a temperature of 90 °C. Then the content of flask was diluted by hot water (50–70 °C) in 3–5 times to decrease acid concentration and stop reaction. After that, mixture was filtered through two paper filters. Holocellulose was first rinsed by hot water to the absence of hydrogen peroxide. Then it was treated with 1:1 alcohol-acetone mixture at the room temperature. Drying of holocellulose was carried out in a vacuum oven at a temperature below 60 °C or in the air to prevent decomposition of hemicelluloses. Holocellulose content in plant raw material was defined by weight method relatively to air-dry sample. Obtaining of holocellulose and determination of holocellulose content in plant raw materials is very important. It allows performing of hemicelluloses extraction better from holocellulose than from plant raw mate-

rial. The value of the holocellulose content in plant material is also important for constructing new lignin-carbohydrate diagram, which will be announced later.

4. 3. Pulping

Cooking of pulp from sunflower stalks by neutral-sulphite (N-Su), soda and alkaline sulphite-anthraquinone-ethanol (ASAE) methods was performed in steel autoclaves of 0.5 dm³ capacity placed in glycerin bath heated to set temperature. The autoclaves were cooled after cooking. Obtained pulp was washed from liquor with running water to neutral pH and dewatered by pressing to dryness about 30 %. Pulp yield was determined by gravimetric method after drying at a temperature of 105 °C during 24 h. Residual lignin content in pulp was determined in relation to absolutely dry raw material (a.d.r.m.) according to existing TAPPI standard procedures [27]. Neutral-sulphite cooking of sunflower stalks was performed by cooking liquor with SO₂ concentration 30 g/l, at a temperature of 175 °C, duration from 30 to 120 minutes, liquid-to-solid ratio 5:1, and pH=9,2. Soda cooking was performed with NaOH consumption 14 % from a.d.r.m. mass, at a temperature of 175 °C, duration from 90 to 180 minutes, liquid-to-solid ratio – 5:1. Alkaline sulphite-anthraquinone-ethanol cooking was performed by sodium sulphite solution and sodium hydroxide with ratio 80:20 volume %, their consumption 25 % from a.d.r.m. mass, alcohol to water ratio 35:65 volume %, anthraquinone consumption 0.1 % from a.d.r.m. mass. Temperature of delignification was 175 °C, duration 30-120 minutes, liquid-to-solid ratio – 5:1. These values of technological characteristics were taken as optimal on basis of previously performed investigations [28]. Cooking by mixture of hydrogen peroxide and acetic acid (peracetic method) was performed in glass flasks with volume 750 ml, which were connected with return condensers. Cooking was performed in the water bath by cooking solution with 35 % hydrogen peroxide solution and 30 % acetic acid solution with ratio 1:1, at the temperature 90 °C, duration from 30 to 120 minutes. Sodium tungstate was used as catalyst with consumption 2 % from a.d.r.m. mass. Liquid-to-solid ratio was 15:1. These values of technological characteristics were taken as recommended on basis of previously performed investigations [29].

To obtain more precise and acceptable experimental data, the pulping of sunflower stalks was performed twice. After the cooking processes, the obtained pulp was refined, thoroughly washed, screened with a 10-mesh sieve and pulp properties were carefully measured.

4. 4. Hand sheet preparation and testing

The pulp samples obtained from sunflower stalks were beaten in centrifugal beating apparatus to reach 60 ± 2 °SR beating degree, determined by the Schopper-Riegler method according to ISO 5267-1 standard. Laboratory hand sheets from obtained pulps with mass of 1 m² 65 ± 1 g were produced in a Rapid Köthen machine in accordance with relevant ISO standard 5269-2 method. Then their strength properties were evaluated according to TAPPI standard procedures: base weight – by T 410 om-02, breaking length – by T 404 cm-92, burst index –

by T 403 om-02, tearing index – by T 414 om-04. Samples were stored at the temperature 20 °C and relative air humidity 65 % during 24 hours before the tests. For every strength property of hand sheets were determined at least 10 times.

5. Results and Discussion

5.1. Chemical composition of annual plants

Chemical composition of sunflower stalks in comparison with the most common representatives of annual plants shows in Table 1.

Table 1

Chemical composition of annual plants, %

Components	Sun-flower	Corn	Wheat straw	Rape-seed	Sun-flower ^a	Corn ^a	Wheat straw ^b	Rape-seed ^b
Holocellulose	67,32	70,15	74,68	62,24	66,85	69,92	74,5	58,51
Cellulose	41,83	42,64	46,27	35,63	47,80	55,70	–	–
Pentosanes	24,36	25,66	26,44	25,57	–	–	–	–
Lignin	20,12	17,98	18,62	22,91	14,43	18,16	15,3	15,52
Extractives								
Ash	3,07	4,74	4,28	3,37	7,99	7,75	4,7	8,80
Hot water	5,68	14,88	10,14	11,64	24,26	16,82	13,99	13,35
Ethanol-benzene	2,15	3,57	5,23	4,82	7,48	8,57	–	–
1 % NaOH	35,57	39,62	38,48	35,61	50,05	46,43	40,59	34,9

Notes: ^a – Ates et al [10]; ^b – Potucek and Milichovsky [16]

According to the data of Table 1, investigated sunflower stalks contain more lignin compared to other represented plants but is close by cellulose and pentosanes content and lesser minerals and water-soluble and alcohol-benzene extractives. Determined chemical composition of sunflower stalks and the most common representatives of agriculture annual plants (wheat straw, corn and rapeseed stalks) is close to found in the literature [10, 16]. Stalks of sunflower as well as wheat straw, corn and rapeseed have higher ash content than hardwood and softwood, but the content of hot water extractives and content of ethanol-benzene extractives of sunflower stalks are close to hardwood and softwood extractives [19]. However, 1 % NaOH extractives content of sunflower stalks, wheat straw, corn and rapeseed stalks is almost twice higher than for softwood and hardwood. Therefore, a priori, it can be assumed that at cooking in an alkaline medium pulp yield from annual plant materials will be lower than from wood and the consumption of chemicals will be higher. Nevertheless, sunflower stalks have less lignin and higher pentosanes

content in comparison with hardwood and softwood. Thus, it can be propose that by their chemical composition the sunflower stalks are suitable to obtain pulp for paper and board production.

As can be seen from Table 1, holocellulose content in the studied representatives of non-wood plant raw materials is slightly higher than the published data. This difference is caused by using the modified technique for determination of holocellulose in plant material. The essence of the proposed modification is reaching low residual lignin content with minimal polysaccharides loss, hydrolytic and oxidizing degradation of cellulose. This is achieved by reducing the time of processing of non-wood plant raw materials by solution of peracetic acid for 30 minutes and shows the following results. Results of studies to determine the residual lignin and holocellulose content in sunflower stalks and other crops (wheat straw corn and rapeseed stalks) depending on the time of plant treatment with peracetic acid solutions shown in Figs. 1, 2.

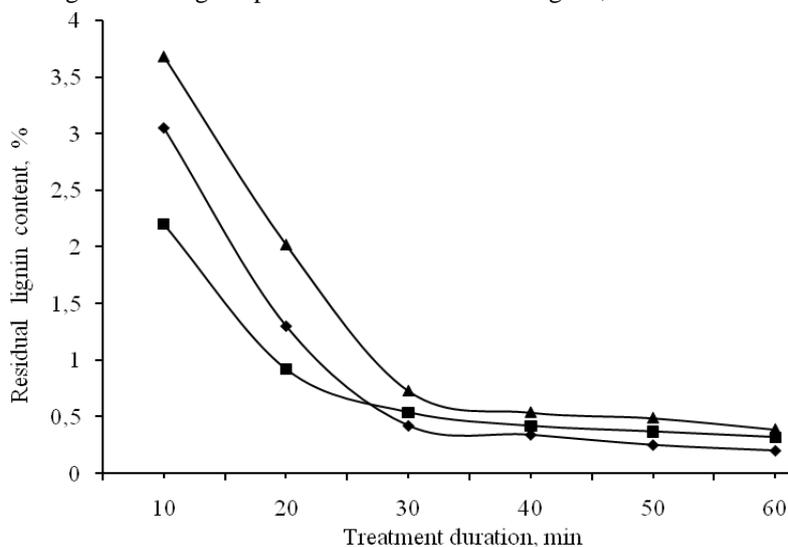


Fig. 1. Dependence of residual lignin content on treatment time of peracetic acid for non-wood plant materials: sunflower (▲), wheat straw (◆) and corn (■)

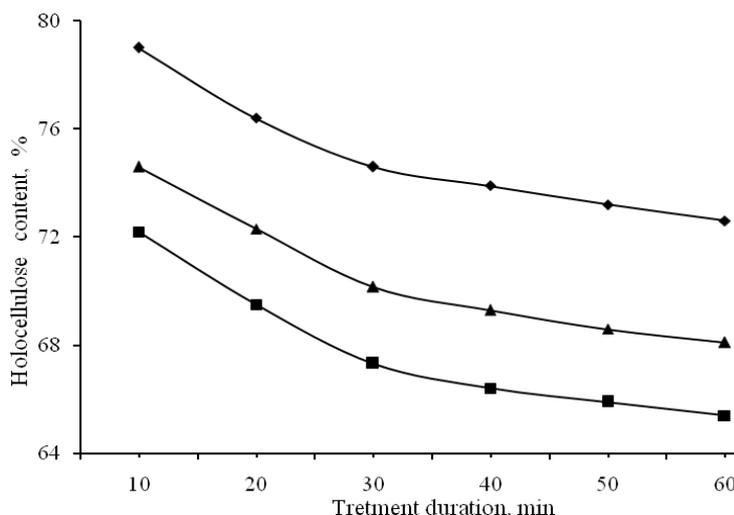


Fig. 2. Dependence of holocellulose content on treatment time of peracetic acid for non-wood plant materials: wheat straw (♦), corn (▲) and sunflower (■)

It is seen from Fig. 1 the residual lignin content in non-wood materials sharply decreases after first 30 minutes of treatment with peracetic acid and then decreases slowly. After 30 minutes treatment of annual plants, residual lignin content remains almost constant, when the holocellulose content continues decreases (Fig. 2). Intensive reduction of holocellulose content is caused by the dominance of polysaccharides cellulose hydrolysis and insignificant removal of the residual lignin from the plant material. Also, another criterion of sufficient treatment was holocellulose color change. After the series of experiments with the different non-wood samples, it was found that holocellulose changes its color from yellow to white after the 30 minutes of treatment with peracetic acid. This proves almost complete absence of lignin in holocellulose and, more precisely, absence of chromophoric groups (carbonyl and carboxyl groups, aromatic rings), contained in lignin and other extractive substances. This is a reason why a further treatment of plant raw material over 30 minutes leads to holocellulose hydrolysis and decreases of its proper value.

Thus, value of holocellulose content in sunflower stalks is 67.32 % from a.d.r.m., which is close to value in literature 66.85 % and was calculated for total sum of 100 % [16]. Value of holocellulose content in sunflower stalks as well as in others annual plants (wheat straw, corn and rapeseed stalks) slightly more than values determined by Wise's method [10].

Advantages of this method of determining holocellulose content in non-wood plant material by solution of peracetic acid is ecologically safer compared to chlorination method. It is based on alternating treatments of deresined raw materials in the presence of water and a solution of organic base in an organic solvent, and also requires lesser duration of experiment and less danger of explosion, than at chlorite (Wise's) method.

5. 2. Pulp properties

To obtain strength properties of pulp from sunflower stalks, series of cooking by neutral-sulphite, soda, and alkaline sulphite-anthraquinone-ethanol and peracetic delignification methods was carried out. Dependen-

cies of yield, residual lignin, physical and mechanical characteristics of resulting pulps on cooking time are demonstrated in Table 2.

It has been observed that increase in cooking time logically decreases pulp yield and residual lignin content for all studied delignification methods. It should be mentioned that the least residual lignin content at the equal yield is observed for ASAE pulp. It can be explained by the fact that during alkaline-organosolv cooking lignin destruction is carried out with breaking of α - and β -ether bonds through intermediate quinonemethyd structure, which is blocked by organic solvent and prevents lignin condensation. Alcohol alkylation of hydroxyl groups in α -position facilitates lignin fragmentation by improving its solubility in organic solvent.

The strength properties of obtained pulps are increasing with the increase of cooking time, which is explained by better papermaking properties of pulp due to additional removing of lignin and extractives and formation of new hydrogen bonds between molecules of cellulose and hemicelluloses. It should be noted that the best strength properties are observed in ASAE pulp in comparison with another studied methods obtaining pulps. It is caused by better protection of pro-xylane against destruction during organosolv cooking allowing it to form stronger matrix in cellulose and, consequently, improve physical and mechanical properties (especially tearing index).

The diagram presented in Fig. 3 confirms such comparative dependence of strength properties of pulps obtained from sunflower stalks by researched delignification methods. The proposed methodology for constructing diagram differs from the known lignin-carbohydrate diagrams Ross, Geertz and Schmidt simplicity of construction, the essence of which is consists of the following.

On the y-axis the pulp yield is indicated from 30 % (for better visualization on the few percent lesser than cellulose content is in the plant raw material) to 100 %. On the y-axis the point corresponding to holocellulose content is also indicated. On the x-axis the percentage value of the lignin content in pulp is indicated from zero to maximum value in plant raw material (for example, 20 % for sunflower stalks). The intersection of horizontal axis at

100 % yield and vertical axis of lignin content creates the point corresponding to initial composition of all plant components (cellulose, hemicelluloses, lignin, resins, fats, and waxes, mineral and others extractive substances). The line, which links this point with the point of holocellulose content in plant raw material, can be considered as the line of “ideal delignification”. It characterizes maximal polysac-

charide content for certain residual lignin content in pulp. Further on lignin-carbohydrate diagram are applied dependences yield on residual lignin content in the pulps obtained by different methods. So the closer the line of certain delignification method is to the line of “ideal delignification”, the higher is polysaccharide yield in the obtained pulp and thus delignification method is more efficient.

Table 2

Parameters of cooking and strength properties of pulps from sunflower stalks obtained by different methods of delignification

Delignification method (Temperature, °C)	Cooking time, min	Yield of pulp, %	Residual lignin, %	Brea-king length, m	Burst index, kN/g	Tear index, mN·m ² /g
N-Su (175)	30	55,8	8,2	3580	2,3	1,3
	60	51,7	6,9	4600	3,1	1,5
	90	47,9	6,4	5160	4,4	2,5
	120	47,1	5,9	5250	5,3	2,7
Soda (175)	90	55,9	11,9	2890	2,4	1,0
	120	52,5	9,6	3450	2,7	1,2
	150	50,9	9,3	3620	3,0	1,3
	180	49,6	8,4	3910	3,1	1,4
ASAE (175)	30	58,1	3,1	4750	4,5	5,4
	60	51,9	2,5	6350	4,8	5,7
	90	48,1	1,6	6850	5,7	5,9
	120	46,9	1,2	7500	6,3	7,1
Peracetic (90)	30	57,5	7,7	3640	1,8	1,0
	60	47,9	6,4	4530	1,9	2,4
	90	42,8	4,6	4740	2,1	2,5
	120	40,2	3,4	5130	2,4	3,0

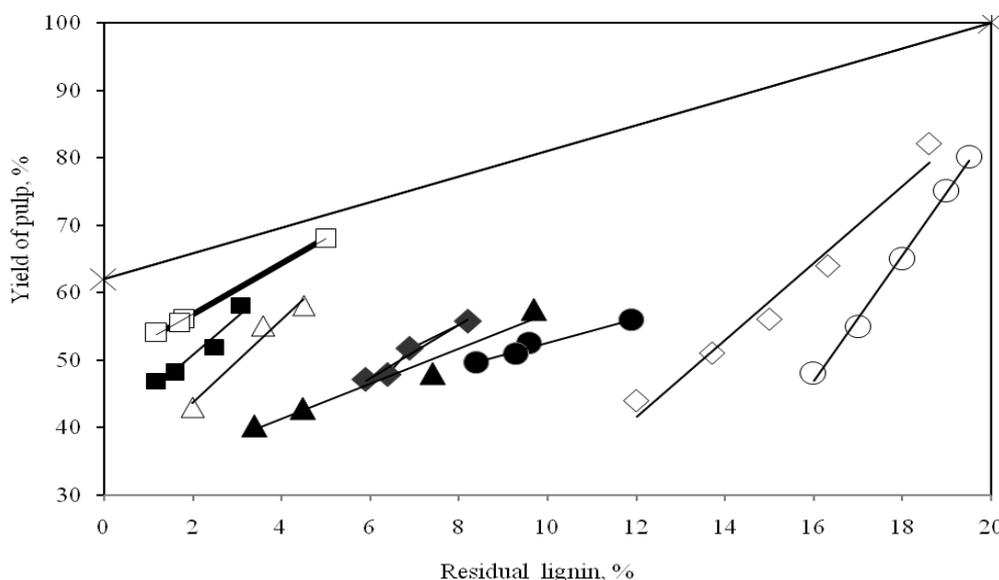


Fig. 3. The lignin-carbohydrate diagram of sunflower stalks delignification by the selected methods: N-Su (◆), soda (●), peracetic (▲), ASAE (■); line of “ideal delignification” (*) and for comparison – delignification of wheat straw by organosolv: ASAE (□), Ester (◇), Acetic (○), peracetic (△)

The dependencies shown on the diagram (Fig. 3) allowed concluding that investigated delignification methods with approaching to the line of “ideal delignification”, i. e. with the increased efficiency of obtaining pulp from sunflower stalks, can be located in following sequence:

$$\text{Soda} \text{ — peracetic — N-Su — ASAE.} \quad (1)$$

In order to compare dependencies of yield from residual lignin content in wheat straw pulp obtained by

ASAE, peracetic, ester ($\text{CH}_3\text{COOH}:\text{CH}_3\text{COOC}_2\text{H}_5:\text{H}_2\text{O} = 33:33:33$ v. %) and acetic ($\text{CH}_3\text{COOH}:\text{H}_2\text{O} = 75:25$ v. %) methods were shown in the Fig. 3. As it is seen in the Fig. 3, ester and acetic methods are significantly inferior to ASAE and peracetic methods by their efficiency removal lignin from plant raw material (Fig. 3). That is why they were not used for obtaining pulps from sunflower stalks. It can be concluded from Fig. 3 that ASAE method is the most efficient among the studied

methods and the pulp is more effectively obtained from wheat straw than from the sunflower stalks, as yield of wheat straw pulp is higher at the same residual lignin content.

The obtained dependence (1) is confirmed by comparison of delignification degree (DD) values calculated for investigated delignification methods by the following equation [30]:

$$DD = 100 - \frac{Y \cdot C}{A}, \% , \quad (2)$$

where A – original lignin content in plant, %; Y – yield of plant residue, %; C – residual lignin content in pulp, %.

The dependencies of delignification degree on cooking time for different delignification methods of sunflower stalks are shown on Fig. 4.

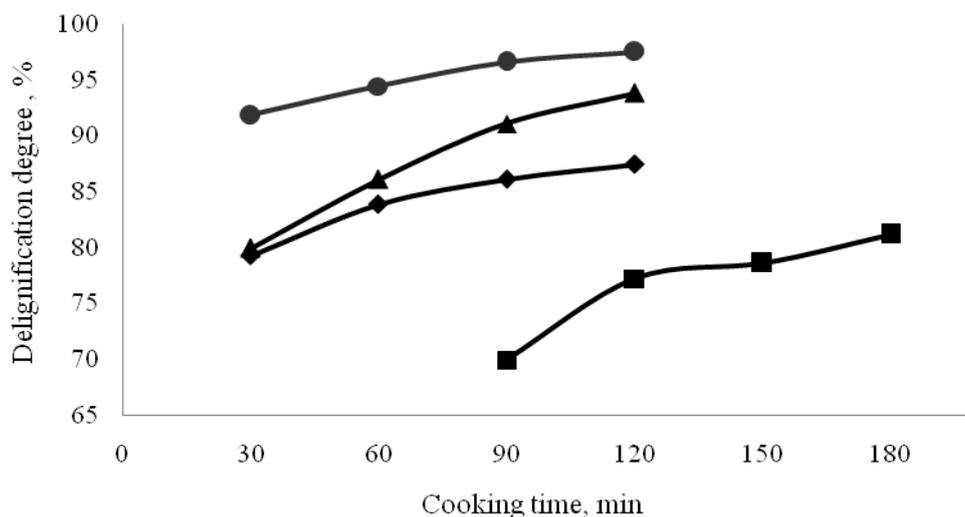


Fig. 4. Dependencies of delignification degree on cooking time of sunflower stalks delignification by different methods: ASAE (●), peracetic (▲), N-Su (◆) and soda (■)

According to obtained dependencies, increasing of cooking time increases delignification degree for all studied methods of delignification raw material, which is consistent with the known concepts of cooking processes theory. It should be noted that alkaline sulphite-anthraquinone-ethanol method is characterized by the highest indices of lignin removal from sunflower stalks and confirms the location of the studied delignification methods of sunflower stalks to obtain sequence (1).

6. Conclusions

The procedure of holocellulose content determination in non-wood plant raw materials was developed. For the minimization of environmental pollution by harmful chemicals and hydrolytic polysaccharides destruction of non-wood plant raw material the treatment by solution of 10 % peracetic acid at a temperature of 90 °C for 30 minutes was recommended. Holocellulose content in non-wood plant material for construction of "ideal delignification" line on lignin-carbon diagram was used. The new more simple methodology of lignin-carbohydrate diagram construction was proposed. Dependencies of yields pulp on residual lignin for studied methods of delignification sunflower stalks, as it approaches to the line of "ideal delignification", are located in the following sequence: soda – peracetic – neutral-sulphite – alkaline sulphite-anthraquinone-ethanol. Dependencies of strength properties of obtained pulps and delignification degrees studied delignification methods of sunflower stalks on cooking time was confirmed obtaining sequence.

It can be concluded from proposed lignin-carbohydrate diagram that delignification of wheat straw is more efficient than from the sunflower stalks at same cooking condition because yield of wheat straw pulp is

higher at the same residual lignin content. Proposed lignin-carbohydrate diagram can be used for comparison as different delignification methods as for one plant raw material, as for different plant raw materials by one delignification method.

Based on the comparative results obtained in this work it can be concluded that sunflower stalks can be a potential source of raw material for pulp and paper industry and among the studied methods ASAE method of delignification sunflower stalks is the most efficient.

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