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THE QUALITY OF THE PULP WASHING PRODUCED BY THE SULPHATE PROCESS IN THE CELLULOSE AND PAPER FACTORY "NATRON HAYAT", MAGLAJ

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ABSTRACT
The process of the pulp washing is very important both economically and environmentally, since the quality of cellulose washing is directly related to the efficiency of chemical regeneration and the quality of the wastewater generated. The purpose of the cellulose fiber washing process is to economically remove the maximum of soluble organic and soluble inorganic substances with a minimum of fresh and recycled water. The filtrate formed during the washing of the pulp contains the lost che
micals during the cooking process and is sent to the regeneration system where the necessary active alkalis are extracted for cooking the pulp and the residual organic matter is burned in an alkaline boiler to obtain the energy required. Poorly washed pulp will carry with it a considerable amount of active alkali, which will end up in the wastewater after the process of thickening the cellulose mass and separating the filtrate. The aim of this paper was to examine the influence of process parameters on the pulp washing quality and determine the quality of the pulp washing in the cellulose and paper factory, Natron Hayat, Maglaj.

1. INTRODUCTION

The pulp after cooking is a suspension of fibers in a waste liquor containing 4-6 m^3 of alkali per tonne of air-dried pulp. In addition to waste liquor, the pulp contains pieces of uncured wood, knots, bark parts, mechanical admixtures, as well as a good deal of shredded fiber. In order for the pulp to be of good quality and to serve its purpose, it must be separated from the impurities and then washed with residual alkali. The impurities present not only spoil the appearance of the technical pulp, but complicate its further processing.

The purpose of cellulose washing is to remove the maximum amount of black liquor and unwanted solutes by using minimum amount of water. The more alkali that goes with the cellulose fiber, the less steam and heat will be produced by the process of evaporation and burning of the alkali. The loss of organic matter of the black liquor in the washing process reduces the organic matter required to produce steam in the alkaline boiler, which will then end up in the wastewater. Also, it will adversely affect the alkali regeneration process, since it will be necessary to add new quantities of alkali to the system to make up for losses, and the lost alkalis, along with other impurities that remain in the pulp will end up partly in the wastewater and affect their quality.There are several ways of washing cellulose mass, and Figure 1 shows a schematic representation of the process of separation and washing of cellulose mass in the factory "Natron Hayat, Maglaj.

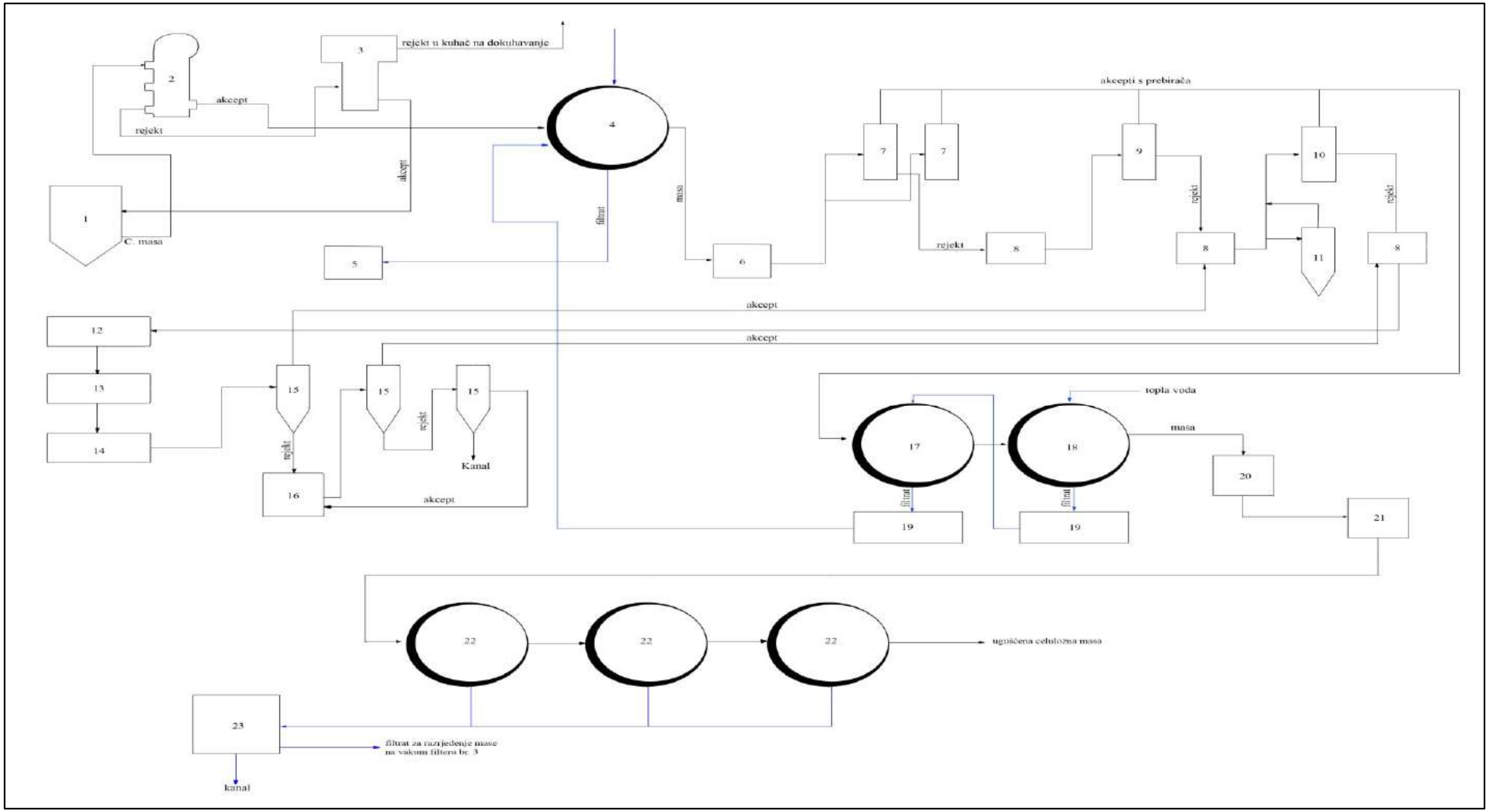

Figure 1. Schematic diagram of the process of separation and washing of cellulose mass: 1-Blow tank, 2-primary knotter, 3-secondary knotter, 4-Rauma repola, 5-pot for filtrate going into the cooker, 6-tank for mass, 7- primary pickers, 8-reek receptacle, 9-secondary picker, 10-tertiary picker, 11-centricliner, 12-press, 13-mill, 14-grinder, 15-centriklinerx battery, 16-rejector, 17 - vacuum filter No.2, 18 -vacuum filter No.3, 19- filtrate tank, 20-stand pipe, 21-mass tank, 22-twin roll presses, 23- return water tank [1]

The loss of alkali calculated as Na_2SO_4 normally ranges from 10-12 kg/t of air-dry (a.d.) cellulose [3]. The regeneration process of the chemical is carried out by evaporating the rare black liquor that is extracted from the cooker and injected into the combustion chamber of the boiler via sprinklers. A reduction reaction takes place in an alkaline boiler [3]:

$$Na_2SO_4 + 2C \rightarrow Na_2S + 2CO_2 \qquad (1)$$

The organic matter is burned, the heat used for steam production is released, some of which is used for energy production, the other part is used in other parts of the plant, and the recovered alkalis from the boiler furnace are discharged into a special container of solvent solution where they are mixed with the waste caustic liqour and a green liqour is formed. Then the green liquor is taken to the process of caustification, where a caustification reaction takes place [2]:

$$Ca(OH)_2 + Na_2CO_3 \rightarrow CaCO_3 + 2NaOH \qquad (2)$$

After that, the decantation process, separation of white liquor from $CaCO_3$, takes place. The resulting $CaCO_3$ is introduced into a rotary kiln, whereby the calcination process takes place. The resulting CaO is further used for the caustification reaction.

2. EXPERIMENTAL PART

The experiment flow consisted of the following steps:

1. Monitoring process parameters to determine their influence on the quality of pulp washing. For a period of six months for each working day from 07:00 to 15:00:

   - the speed of the dosing wheel that leaks the feedstock was monitored, and based on the number of the dosing wheel rotation, the capacity of the produced pulp (C.P.P.) for that day, expressed as tonnes of produced cellulose/day, wascalculated:

$$\text{C.P.P.} = \frac{\text{rotation number of dosing wheel} \cdot 60 \cdot 24 \cdot 0{,}31 \cdot 0{,}145 \cdot 0{,}48}{0{,}9} \left(\frac{\text{tonnes}}{\text{day}}\right) \qquad (3)$$

2. White liqour samples were taken. The white liquor is used for cooking wood raw material and .the formation of cellulose fibers. For the sulphate process it consists of NaOH and Na_2S. The content of active alkali (concentrations) by volumetric (titration) method (expressed as g/dm^3NaOH) was analyzed in white liquor and cellulose samples were taken after the cooking process. Kappa number was also analyzed. Kappa number is an indicator of the content of lignin in the pulp or an indicator of the hardness of the pulp.

3. Testing the parameters that define the quality of the washed pulp. For six months every working day at 09:00 and at 13:00 h, after the pulp washing process, samples of washed pulp were taken and:
   - cellulose mass concentration (%) was analyzed
   - alkali loss was determined in three ways:
   - loss of alkali calculated as kg Na_2SO_4 / $t_{a.d.}$ cellulose - residual alkali in the pulp,
   - loss of alkali calculated as kg Na_2SO_4 / $t_{a.d.}$ cellulose - residual alkali in the filtrate obtained by squeezing the cellulose mass and
   - loss of alkali in mass determined and calculated as g/dm^3 Na^+.

After the washing process, the washed cellulose mass is thickened by means of presses, and the resulting filtrate is actually wastewater that goes into the wastewater collection channel leading to the wastewater treatment plant.

4. Testing the parameters that define the quality of the generated wastewate. For a period of six months, samples of waste water generated after pressing the washed cellulose mass (filtrate) were taken every working day and the following parameters were analyzed:
   - loss of alkali in wastewater expressed as g/dm^3 Na^+,
   - electrical conductivity,
   - pH value,
   - chemical oxygen demand, COD,
   - biological oxygen demand, BOD_5.

Wastewater generated after washing and pressing the pulp together with wastewater from other parts of the factory is collected and sent through the sewage canal to the wastewater treatment plant.

2.1. Analyzed parameters and test methods

All parameters were analyzed according to the internal methods of the Natron Hayat factory, Maglaj [1]. The sampling site and the pulp sample after the Kamyr cooker for analysis and determination of the Kappa number are shown in Figure 2.

Figure 2. Sampling site and the pulp sample [1]

The sample of washed cellulose is taken from vacuum filter no. 3, from the last stage of washing and is removed with a shovel and placed in a container. The sampling site and the washed cellulose sample for analysis are shown in Figure 3.

Figure 3. Sampling site and washed cellulose sample [1]

The sampling point of the filtrate, the wastewater that is generated after pressing the washed cellulose mass and the collecting duct, are shown in Figure 4.

Figure 4. Wastewater effluent resulting from the pressing of washed cellulose [1]

3. RESULTS AND DISCUSSION

Table 1 shows the average values of the process parameters for each month during the observed period.

Table 1. Average values of process parameters for each month in the observed period[1]

Month	Kappa number	Active alkali (g/dm^3 NaOH)	Production (t/dan)
october	42,452	130,505	286,589
november	41,878	132,191	282,771
december	42,479	129,596	274,597
january	41,014	130,409	269,856
february	42,900	130,174	260,601
april	45,445	126,896	285,128

Table 2 shows the average values of the pulp washing quality control parameters.

Table 2. Average values of pulp washing quality control parameters for each month during the observation period for the number of samples taken [1]

	Parameters determined for the pulp sample from vacuum filter no. 3		
Month	Cellulose mass concentration (%)	AL_1	AL_2
october	11,839	16,371	0,180
november	11,380	17,139	0,168
december	11,038	19,754	0,277
january	11,180	18,461	0,242
february	10,959	16,127	0,122
april	11,579	16,617	0,156

Legend: AL1 - alkali loss in cellulose mass (kg Na_2SO_4/t $_{a.d.}$ cellulose) - internal method; AL2 - alkali loss in cellulose mass (g /dm^3 Na^+ ions) - SCAN 37:98

Table 3 shows the average values of wastewater quality control parameters generated after pressing the washed pulp for each month.

Table 3. Average values of wastewater quality control parameters which is obtained after pressing the washed cellulose mass, and which enters the wastewater channel

	Wastewater parameters which, after pressing the washed cellulose mass, enters the wastewater channel				
Month	***AL_3***	***Electrocondutivity (μS/cm)***	***pH***	***COD (mg/dm^3 O_2)***	***BOD_5 (mg/dm^3 O_2)***
october	0,107	591,103	8,654	2604,235	211,500
november	0,107	639,595	8,541	2493,355	305,833
december	0,156	767,575	8,343	2158,629	337,667
january	0,116	615,077	8,208	1522,750	328,625
february	0,094	530,125	8,713	1659,529	300,571
april	0,106	600,226	8,660	1452,903	109,000

Legend: AL_3- Loss of alkali in the filtrate (wastewater generated after pressing the washed cellulose) (g/ dm^3 Na^+ ions) - SCAN 37:98

It can be seen from Figure 5 that the Kappa number was within the permitted limits of 40 ± 3, except for the month of april when it was 45,445. Production in april amounted to 285,128 t/day and was higher than in other months, while the content of active alkali in white liquor for this month was the lowest, amounting to 126,896 g/dm^3 NaOH. Thus, due to the increased production, and as production is directly proportional to the speed of the metering wheel more raw material was leaked into the Kamyr cooker, and due to the reduced content of active alkalis in the cooking liquid (white liquor), the raw material could not be cooked well. As a result, the Kappa number was increased and a hard cooked pulp was obtained.

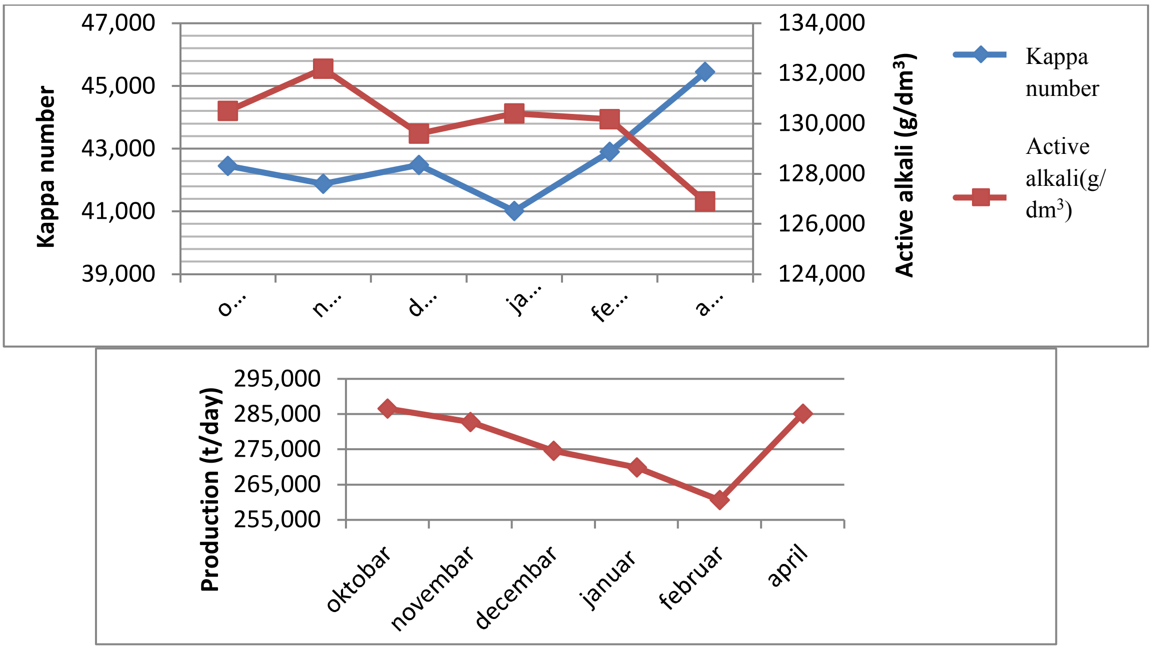

Figure 5. Correlation between production capacity, active alkali content and Kappa number [1]

It can be seen from Figure 6 that the lowest alkali loss was for the months of october, february and april and amounted to about 16 kg of Na_2SO_4/t a.d. cellulose. The month of april is characterized by very high production, but also the highest Kappa number. This means that the mass is not sufficiently cooked, which is hard (higher lignin content), these active alkalis in the white lye did not sufficiently enter the pores of the cellulose chips, and when washing it was necessary to remove all residual bumps and residual alkalis in the the masses due to insufficient cooking are not large, so they are easily removed when washing and better washed pulp is obtained. The highest loss of alkali in the pulpwas in december and amounted about 19,754 $Na_2SO_4/t_{a.d.}$cellulose. February had the lowest production capacity, but consequently the loss of alkali was low and the Kappa number was within the permitted limits.

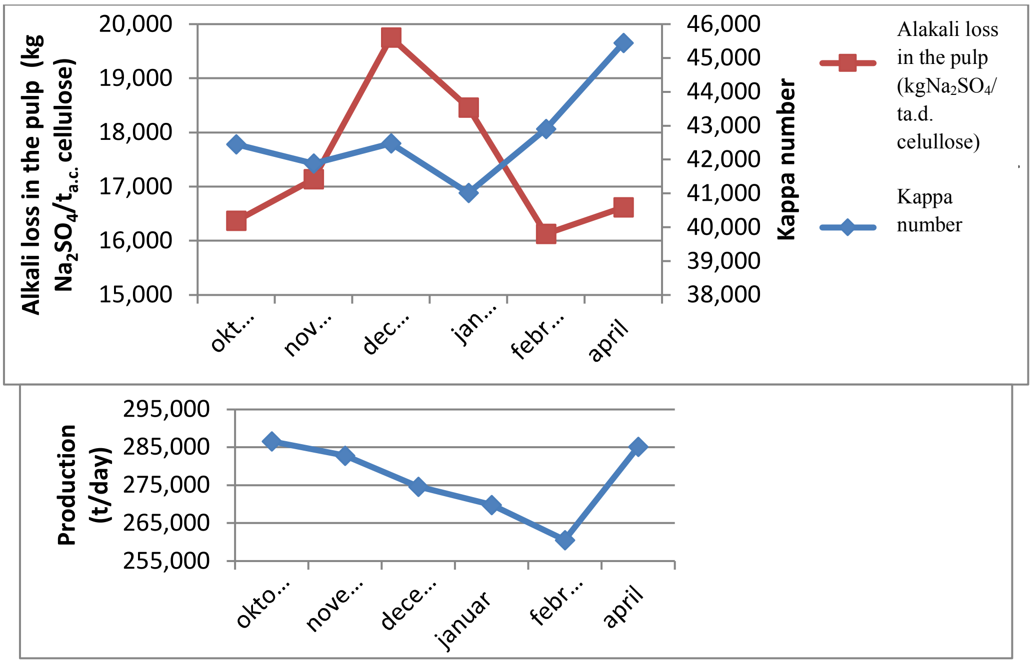

Figure 6. Correlation between production capacity, alkali los in the pulp and Kappa number[1]

It can be concluded that Kappa number and production capacity have an influence on the alkali loss in the mass, but to determine exactly to what extent other process parameters need to be analyzed.

COD, BOD_5, conductivity and pH were analyzed to determine the quality of wastewater generated after pressing the pulp, and thus the quality of the pulp washing.

Figure 7 presents a diagram showing the dependence of the alkali loss in the washed cellulose mass and the alkali loss in the wastewater that is generated after pressing and concentrating the pulp.

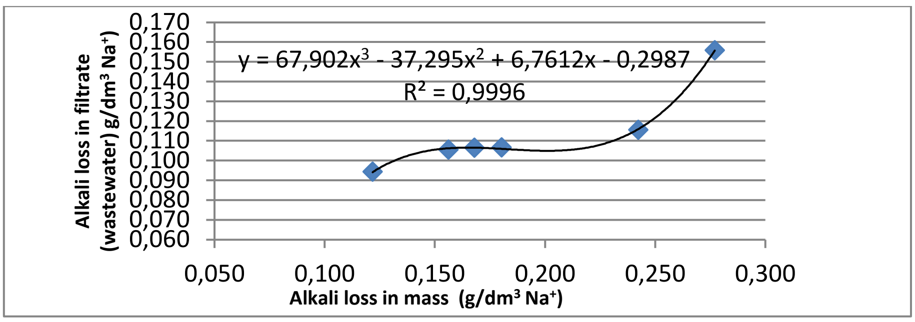

Figure 7. Effect of of alkali loss in the mass on the loss of alkali in wastewater [1]

It can be seen from Figure 7 that the highest alkali loss of 0,277 g/dm^3 Na^+ in the washed cellulose mass corresponds to the highest alkali loss in the generated filtrate of 0,156 g/dm^3

Na^+. Also, the lowest alkali loss in washed cellulose of 0,122 g/dm^3 Na^+ corresponds to the lowest alkali loss in the generated filtrate of 0,094 g/$dm^3$$Na^+$.

Figure 8 shows the effect of alkali loss in the washed cellulose on the electroconductivity of the generated wastewater.

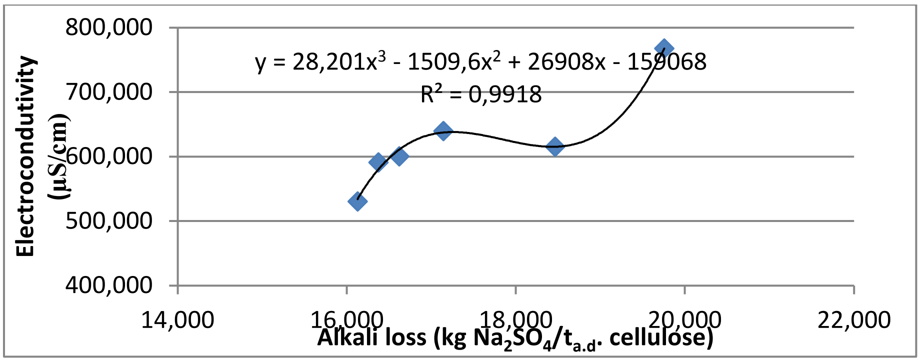

Figure 8. Effect of alkali loss in washed cellulose on the electroconductivity of the generated wastewater [1]

Figure 8 shows that the lowest alkali loss in the washed pulp of 16,127 kg $Na_2SO_4/t_{a.d.}$ cellulose corresponds to the lowest electroconductivity of 530,125 µS/cm. Also, the highest alkali loss of 19,754 kg $Na_2SO_4/t_{a.d}$ celullose corresponds a maximum conductivity of 767,575 µS/cm.

Figure 9 shows the effect of alkali loss in the resulting wastewater on the electrical conductivity of the resulting wastewater (filtrate).

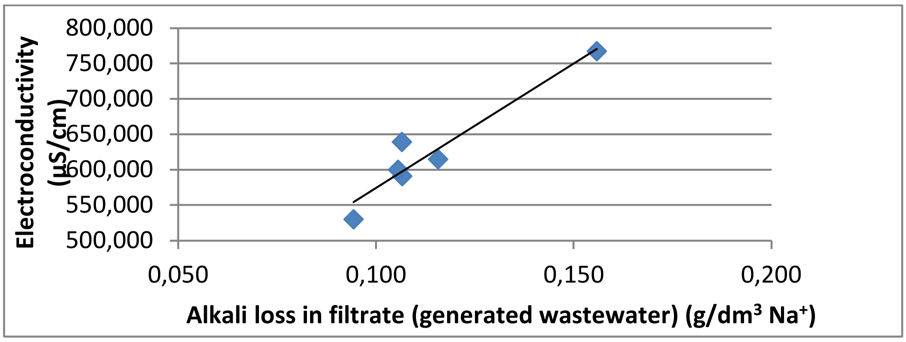

Figure 9. Effect of alkali loss in the filtrate on the electrical conductivity of that filtrate [1]

It can be observed from figure 9 that the maximum conductivity value is 767,575 µS/cm and corresponds to a maximum alkali loss in the generated wastewater that amounts 0,156 g/dm^3 Na^+, while the minimum conductivity value is 530,125 µS/cm and corresponds to a minimum alkali loss of ,094 g/dm^3 Na^+.

The pH value of the generated wastewater (filtrate) after pressing the washed cellulose ranges from 8,2 to 8,7 because it is an alkaline production process, so it is expected that this pH is in the base region.

Washing quality is evaluated on the basis of the alkali loss parameter. If the washing is bad the loss of alkali in the washed mass will be higher. It has been shown that with an increase in the loss of alkali in the washed mass, the loss of alkali in the filtrate, ie the wastewater that is generated after pressingthe washed mass, increases, which ultimately affects the quality of the wastewater. With increasing alkali loss in the resulting wastewater, the conductivity of that water increases.

The quality of generated wastewater after pressing the washed pulp generated was also determined analyzingCOD and BOD_5 parameters. These two parameters of the generated wastewater can also indicate the quality of washing, since worse washing means increased COD and BOD_5 because organic and inorganic matter will be left behind in the mass and increase their values. It has been proven that if the alkali loss in the filtrate is increased then both COD and BOD_5 are increased because this means that the cellulose is poorly washed and more organic matter will be left behind.

Based on the analyzes carried out in the defined research period, the characteristic high and low values of COD of the generated wastewater and the corresponding values of alkali loss were observed (Table 4).

Table4. Characteristic values of COD and alkali loss in the resulting wastewater [1]

COD of generated wastewater ($mg/dm^3\ O_2$)	Alkali loss in generated wastewater ($g/dm^3\ Na^+$)
4105	0,1279
3184	0,109
848	0,0768
4835	0,1861
4720	0,1477
912	0,0814

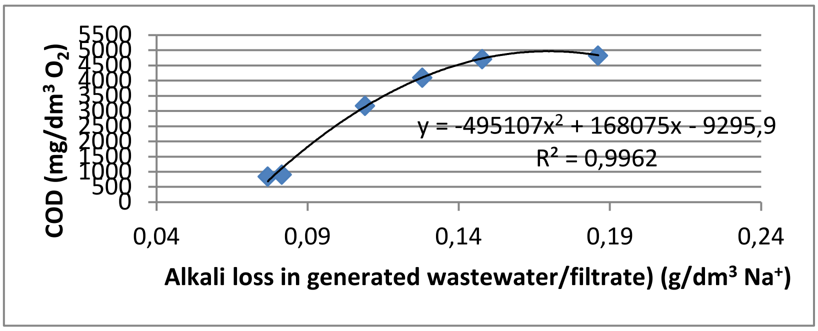

Figure 9 .Correlation between alkali loss in filtrate and COD of filtrate [1]

It can be seen from figure 9 that due to the low values of alkali loss in the resulting wastewater (filtrate), COD values will be low and due to the very high values of alkali loss COD values will be high. If washing is very bad, which means higher alkali loss in the resulting wastewater, there will also be a lot of residual organic matter due to poor washing, which is why COD value is elevated.

From Table 3 it can be seen that the water generated after pressing the washed cellulose is heavily loaded with organic pollution because they have very high values of COD and BOD_5. If the washing is not done well, organic matter will be left behind in the water and this will cause an increase in COD and BOD_5 values.

Figure 10 shows BOD_5 and alkali loss values in the resulting wastewater. From the figure 10 it can be seen that the maximum value of BOD_5 was in the month of december and in the month of december there was also a maximum alkali loss. The minimum BOD_5 value was in april, and the alkali loss was then 0,106 g/dm^3. Based on this, it can be seen that the worse the mass is washed, the greater the loss of alkali in the wastewater is and also it is higher theBOD_5value of the wastewater, since poor washing means a higher content of organic residues.

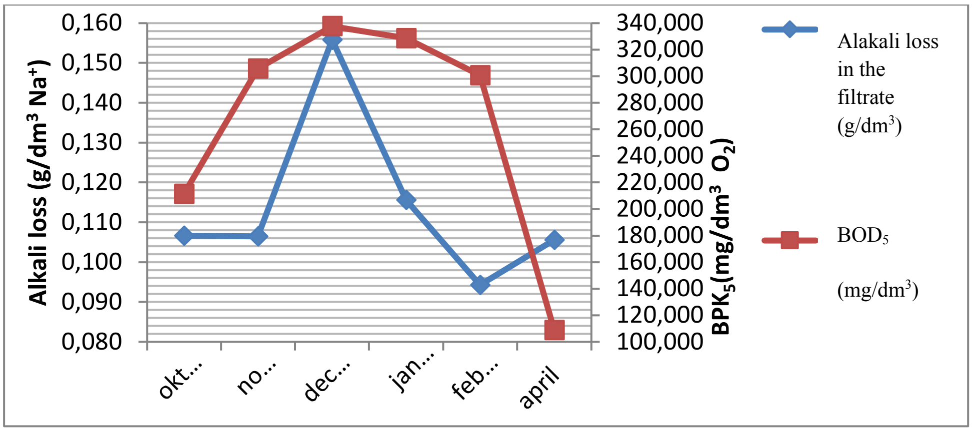

Figure 10. Average values of loss of alkali and BOD_5 of the resulting filtrate for each month in the observed period [1]

4. CONCLUSION

Based on the performed examinationand the obtained results, it can be concluded that the process parameters - production capacity, content of active alkalis in white liquor and kappa number affect the process of washing cellulose mass. The quality of washing expressed through the loss of alkali in the pulp and in the resulting wastewater after pressing the washed pulp is very good. The organic load of the wastewater generated is high and is directly related to the washing of the pulp. It can also be concluded that poor washing of the pulp means an increase in the loss of alkali in the washed mass, which causes an increase in the loss of alkali in the filtrate after pressing the washed pulp. Bad washing also causes an increase in the organic loading of the filtrate. All of the above will ultimately have an adverse effect on the chemical regeneration process and the wastewater treatment process.

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