CHEMICAL PROCESSING OF WASTE WOOD BASED AGGLOMERATES PART I: EVALUATION OF PROPERTIES OF FLUTING LINERS MADE OF SEMICHEMICAL PULP OBTAINED BY A MILDLY ALKALINE SULPHUR-FREE COOKING PROCESS

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ABSTRACT

The article describes the method of evaluation and preparation of fluting liners produced from semichemical pulp of waste wood particle boards (PB) and oriented strand boards (OSB) and after the combination with old corrugated cardboards (OCC) to improve strength properties. The semichemical pulp was obtained by a mildly alkaline boiling process from two fractions of waste PB and OSB. Properties as thickness, bulk density, gurley, tensile strength, tensile index, breaking length, burst index, CMT₃₀ and SCT were monitored on lab sheets 127 gm⁻² and 170 gm⁻². Values of pH and residual NaOH and Na₂CO₃ were determined in batch leachate.

KEYWORDS: Waste PB and OSB, fluting, semichemical pulp, alkaline sulphur-free cooking process, delignification.

INTRODUCTION

Several patents deal with the recycling of wood-based agglomerated materials such as PB or OSB using steam and higher temperatures for the preparation of wood particles for their reuse (DE-AS 1201045 (1963), DE4224629 (1994), DE 10144793 (2003)). The decomposition of waste

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wood damages the environment by harmful degradation products (Rowell et al. 1993, Michanickl 1996, Grigoriou 1996). Recycling of wood waste is difficult due to a content of harmful chemicals contained both in glue used during a manufacture process (Risholm-Sundman and Vestin 2005) and in additives which originally served to protect it from moisture content, wood decaying fungi, to increase fire resistance and so on (Erbreich 2004). Ihnát et al. 2017 deals with the recycling of waste PB and OSB with the emphasis on their reuse for particleboards, other works are focuses on a reuse in medium-density particleboards (Mo et al. 2003), respectively in low density particleboards (Wang and Sun 2002) or to find a suitable use of this material, for example sound absorbing construction (Yang et al. 2003). As well as other lignin-cellulose materials such as straw or waste paper (Ihnát et al., 2015a, b, Lubke et al., 2014), both waste PB and OSB can be used for a fiber production when looking for a cheap replacement for example for a fluting.

The production of semi-chemical pulp (yield of about 80%) by the neutral sulphite cooking process (NSSC) is the best in terms of the pulp quality. Main components of the boiling solution are Na_2SO_3 and Na_2CO_3 in the NSSC cooking process (Olszewski 1972a, b, Farkas et al., 1978, Janci et al 1988, von Koeppen 1986, Keskin 1994, Lovelady 1991, Bierman 1996, Balbercak 1998). This cooking process requires in addition to a primary regeneration also a secondary regeneration of chemicals, which is relatively complicated and it is a major source of air pollution by hydrogen sulphide and other sulphur exhales (Hojnos 1975). A replacement of the NSSC can be a mildly alkaline sulphur-free technology, i.e., technology where Na_2SO_3 is replaced by NaOH in the boiling solution (Balbercak 2002a, b, Lindstrom 2002). The mildly alkaline sulphur-free technology of semi-chemical pulp production is much more gentle to wood mass compared to the other method - the natron batch.

A mixture of hardwood and softwood in ratio of birch 50 % wt. + poplar 30-50 % wt. + hornbeam 0 - 20 % wt. was used to produce a semi-chemical pulp with a yield of 78-82 % with a lignin content of 15-18 % and a degree of grinding of $25-30^{\circ}$ SR in Slovakia.

The composition of the boiling solution was:

- 133.4 140.2 g·l⁻¹ Na₂CO₃ i.e. 78 82 g·l⁻¹ recalculated as Na₂O
- 28.4 23,22 gl⁻¹ NaOH i.e. 22 18 gl⁻¹ recalculated as Na₂O
- ratio Na_2CO_3 : NaOH = 3.55 4.55 as Na_2O
- alkaline content = 6.5 7.0 % Na₂O / o. d. wood

The cooking itself or delignification is divided into three phases and lasted 37.5 - 48 min: Phase I - impregnation at 125°C, which lasted about 2.5 minutes

Phase II - delignification, where the chips were cooked in a boiling solution for 25-35 min at 165-178°C

Phase III - retention time to the desired pulp yield of about 10-15 min with the temperature maintenance

The average observed parameters of 127 g·m⁻² are:

-	CMT ₃₀	220 – 280 N
-	Burst index	2.5 – 3.0 kPa·m ² ·g ⁻¹
-	Tensile strength	6.9 − 7.5 kN·m ⁻¹
-	Tensile index	43,0 − 50.0 N·m·g ⁻¹

MATERIAL AND METHODS

In the chemical processing of waste from PB and OSB a mildly alkaline method of delignification was used, the main components of the boiling solution being NaOH and Na_2CO_3 in a ratio of 1: 4. The waste PB and OSB were treated in two ways for the chemical treatment:

- Sample processed on small cubes with sides about 15mm
- Sample disintegrated and only selected fraction (4-8 mm) used

Preparation of lab sheets from semichemical pulp obtained from fraction 15x15 mm using mildly alkaline sulphur-free cooking process

Cube-shaped samples of about 15x15 mm resembling wood chips were used in the first phase of the mildly alkaline sulphur-free cooking process of waste PB and OSB. The purpose of this test was to obtain initial information on the possibilities of delignification of such waste and information on the basic mechanical properties of the semichemical pulp prepared.

The cooking solution was laboratory prepared from clean chemicals and its composition is shown in Tab. 1.

	Cooking solution
pН	12.1
NaOH (g·1-1)	25.8
Na ₂ CO ₃ (g·1 ⁻¹)	136.8
NaOH as (g·l ⁻¹ Na ₂ O)	20.00
Na_2CO_3 as (g·1 ⁻¹ Na ₂ O)	80.00
Na ₂ O (g·1 ⁻¹)	100.0

Tab. 1: Basic characteristics of the cooking solution.

Laboratory batches were performed in 750 ml lab autoclaves under the same conditions:

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Cooking solution	7-15% as Na_2O / o. d. wood
Hydromodul	4: 1
Impregnation time	10 - 20 min
Impregnation temperature	125°C
Total batch time	20 - 60 min
Batch temperature	165°C

The cooked samples were separated from the leachate after the boiling and washed several times with hot water. The yield was determined and the cooked samples were defibrated in a laboratory mixer for 1 minute. Individual samples were sorted using a laboratory sorter Wewerk on a screen with 0.25 mm slots after the defibering. The substance was pulped on a Valley Laboratory Mill (Pažitný et al. 2011) at 25°SR and 30°SR.

Laboratory sheets weighing 127 and 170 g·m⁻² were prepared from sample of semi-chemical pulp prepared at 30°SR. Thickness, bulk density, gurley, tensile strength, tensile index, breaking length, burst index, CMT_{30} a SCT were measured. Values of pH and residual NaOH and Na₂CO₃ were determined in batch leachate.

Preparation of lab sheets from semichemical pulp obtained from fraction 4-8 mm using mildly alkaline sulphur-free cooking process

The same solution was used as in previous batches (Tab. 1) for the mildly alkaline sulphurfree cooking procedure of semichemical pulp preparation from the 4-8 mm fraction. The impregnation time, the total batch time and the batch temperature were extended due a better impregnation. The batches with the mildly alkaline sulphur-free cooking process of semichemical pulp were carried out under the following conditions:

Content	100 g oven dry
Cooking solution	7 - 20% as Na_2O / absolutely dry wood
NaOH / Na ₂ CO ₃ , g·l ⁻¹ Na ₂ O	20/80
Hydromodul	4: 1
Impregnation time	20 - 60 min
Impregnation temperature	125°C
Total batch time	45 - 120 min
Batch temperature	170°C

The cooked samples were separated from the batch leachate. The material was washed several times with hot water and the yield was determined. The samples then were defibrated in a laboratory mixer. The individual samples were then milled in a Jokro laboratory mill for 30 minutes and subsequently sorted using the Wewerk laboratory sorter on a screen with 0.25 mm slots. The substance was pulped on the Jokro laboratory mill at 35 °SR. Laboratory sheets with a weight of 127 g·m⁻² were prepared and the parameters similar to those in the previous case were determined.

Preparation of lab sheets with OCC addition

Laboratory sheets from semichemical pulp with addition of OCC were prepared from selected pulp samples obtained from both fractions. The addition of OCC ranged between 20-60 %. Parameters as burst index, CMT_{30} a SCT were observed on samples. Strength parameters were compared on lab sheets weighing 127 gm⁻².

RESULTS AND DISCUSSION

The conditions and results of laboratory cooking of cube-shaped samples of waste PB and OSB using the mildly alkaline sulphur-free method for the semichemical pulp preparation are shown in Tab. 2 and Tab. 3. The strength parameters were determined only from sheets weighing 170 g·m⁻², since the required strength parameters could not be determined from sheets weighing 127 g·m⁻² due to their pure strength (Balbercak and Kuna 2002a)

Batch No.	A1/1	A1/2	A1/3	A1/4	A1/5	A1/6
Sample	DTD	DTD	DTD	DTD	DTD	DTD
Impregnation at 125°C	10	10	10	15	15	15
Time of cooking at 165°C	10	20	30	30	30	30
Content of active alkali (% Na ₂ O)	7	7	7	7	10	12
Yield (%)	75.1	73.4	73.4	73.7	71.7	70.4
Content of non-cooked matter (%)	22.1	21.0	20.9	19.6	18.4	17.9
Residual lignin (%)	16.5	16.3	16.4	16.4	16.1	16.1
pH	9.0	8.9	8.9	9.0	9.0	8.9
Residual NaOH (g·1 ⁻¹)	0	0	0	0	0	0
Residual Na ₂ CO ₃ (g·1 ⁻¹)	23.3	24.6	24.7	24.1	34.0	41.2
Weight (g·m ⁻²)	170	170	170	170	170	170
Thickness (µm)	351	330	332	328	330	330
Bulk density (g·cm ⁻³)	0.49	0.52	0.54	0.53	0.51	0.52
Tensile strength (kN·m ⁻¹)	3.00	3.09	3.08	3.09	3.58	3.62
Tensile index (Nm·g ⁻¹)	17.90	17.10	17.90	18.50	19.90	20.70
Breaking length (km)	2.44	2.47	2.40	2.50	2.59	2.65
Burst index (kPam ² ·g ⁻¹)	0.50	0.50	0.50	0.50	0.60	0.60
CMT ₃₀ (N)	-	-	-	-	-	-
SCT (kN·m ⁻¹)	-	-	-	1.10	1.10	1.10
Gurley (s)	6.40	6.40	6.00	7.00	11.00	15.00

Tab. 2: Conditions and results of laboratory cooking of cube-shaped samples of waste PB using the mildly alkaline sulphur-free method.

Tab. 3: Conditions and results of laboratory cooking of cube-shaped samples of waste OSB using the mildly alkaline sulphur-free method

Batch No.	A2/1	A2/2	A2/3	A2/4	A2/5	A2/6
Sample	OSB	OSB	OSB	OSB	OSB	OSB
Impregnation at 125°C	10	10	10	20	20	20
Time of cooking at 165°C	10	20	30	40	40	40
Content of active alkali (% Na ₂ O)	7	7	7	10	12	15
Yield %	85.4	84.1	81.5	80.3	80.1	78.6
Content of non-cooked matter (%)	22.1	22.0	20.8	20.6	20.4	19.9
Residual lignin (%)	18.5	18.3	18.4	18.2	18.1	18.1
pH	9.3	9.0	8.9	8.9	9.2	9.3
Residual NaOH (g·l-1)	0	0	0	0	0	0
Residual Na ₂ CO ₃ (g·1 ⁻¹)	13.6	13.3	12.3	18.7	28.8	39.8
Weight (g·m ⁻²)	170	170	170	170	170	170
Thickness (µm)	321	320	312	310	305	328
Bulk density (g·cm ⁻³)	0.59	0.58	0.56	0.56	0.55	0.54
Tensile strength (kN·m ⁻¹)	3.00	3.09	3.08	4.74	4.40	4.46
Tensile index (Nm·g ⁻¹)	17.90	17.10	17.90	27.10	25.00	25.20
Breaking length (km)	2.44	2.47	2.40	3.50	3.39	3.55
Burst index (kPam ² ·g ⁻¹)	0.50	0.50	0.50	0.80	0.80	0.80

CMT ₃₀ (N)	-	_	_	94	94	96
SCT (kN.m)	-	-	-	1.10	1.20	1.20
Gurley (s)	6.40	6.40	6.00	31.00	36.00	29.00

Under the same batch conditions, about 10% lower yields of semichemical pulp from PB were achieved compared to OSB. The yields from the PB cube-shaped samples were also lower compared to the yields of the semichemical pulp produced in Sturovo (Slovakia) from the mixture of different species of deciduous wood (Balbercak and Kuna 2002b). Large differences can be caused by a different content of adhesives and coatings in individual types of waste (Ihnát et al. 2017). An extending the cooking time and increasing the demand for cooking chemicals resulted in a deeper cooking, as evidenced by the lower yields for both PB and OSB waste, as well as a slight decrease in a residual lignin in semichemical pulp (Janci 1988).

The high content of a non-cooked matter, which ranged from 17.9 to 22.1 % at yields of 70.4 - 85.4 % for PB and OSB waste, says about a poor impregnation of cube-shaped samples (Bierman 1996). This may be due to a high content of cured adhesives among wood particles, but also inside of wood particles.

From the evaluation of the mechanical properties determined on the 170 g·m⁻² laboratory sheets, we can see that better strength parameters were achieved in case of semichemical pulp prepared from OSB waste at the yield of 78.6-80.3 %, compared to the PB waste. Burst index, CMT_{30} and SCT are the most important parameters from the point of view for the semichemical pulp using for fluting or liners. As shown in Tab. 2 and Tab. 3 nor the CMT_{30} was able to determine due a samples cracking from majority of the semichemical pulp prepared. Burst index values were very low, up to several times, compared to other researches (Pažitný et al. 2013). Under the above mentioned conditions, the highest values of the burst index equal 0.8 kPam²·g⁻¹ were achieved in semichemical pulp prepared from the OSB waste in the range of yields between 78.6-80.3 %.

The results of cooking shown in Tab. 2 and Tab. 3 show that under conditions of mildly alkaline sulphur-free technology close to the conditions used in the production of fluting in Sturovo (Balbercak and Kuna 2002) the cube-shaped PB and OSB waste with a size of 1.5 x 1.5 cm a semichemical pulp can be prepared but strength parameters are relatively low and did not correspond to 30 % of required values. Such prepared semichemical pulp is not suitable for the production of even the least quality liners.

Batch No.	A4/1	A4/2	A4/3	A4/4	A4/5	A4/6
Sample	DTD	DTD	DTD	DTD	DTD	DTD
Impregnation at 125°C	20	20	20	60	60	60
Time of cooking at 165°C	25	35	45	20	40	60
Content of active alkali (% Na ₂ O)	7	7	7	20	20	20
Yield (%)	78.5	76.0	74.0	75.4	74.1	73.2
Content of non-cooked matter (%)	19.1	19.0	18.9	14.6	14.4	13.9
Residual lignin (%)	15.5	14.3	13.4	13.8	13.4	13.3
pH	7.9	7.9	7.8	8.2	8.2	8.1
Residual NaOH (g·1-1)	0	0	0	0	0	0
Residual Na ₂ CO ₃ (g·1 ⁻¹)	21.2	15.9	9.3	58.3	58.3	58.1

Tab.4: Conditions and results of laboratory cooking of the PB disintegrated waste using the mildly alkaline sulphur-free method.

Weight (g·m ⁻²)	127	127	127	127	127	127
Thickness (µm)	255	251	250	270	267	267
Bulk density (g·cm ⁻³)	0.53	0.50	0.50	0.50	0.49	0.49
Tensile strength (kN·m ⁻¹)	2.30	2.39	2.41	2.50	2.61	2.63
Tensile index (Nm·g ⁻¹)	18.90	19.10	19.30	19.50	19.90	20.10
Breaking length (km)	2.44	2.47	2.50	2.53	2.58	2.60
Burst index (kPam ² ·g ⁻¹)	0.60	0.60	0.60	0.70	0.80	0.80
CMT ₃₀ (N)	90.00	94.50	94.00	100.0	104.00	104.00
SCT (kN·m ⁻¹)	1.10	1.10	1.10	1.10	1.20	1.20
Gurley (s)	1.40	1.40	1.60	1.70	1.70	1.90

Tab.5: Conditions and results of laboratory cooking of the OSB disintegrated waste using the mildly alkaline sulphur-free method.

Batch No.	A4/7	A4/8	A4/9	A4/10	A4/11	A4/12
Sample	OSB	OSB	OSB	OSB	OSB	OSB
Impregnation at 125°C	20	20	20	60	60	60
Time of cooking at 165°C	25	35	45	20	40	60
Content of active alkali (% Na ₂ O)	7	7	7	20	20	20
Yield (%)	84.8	84.0	80.1	79.4	78.3	76.6
Content of non-cooked matter (%)	21.1	20.5	20.2	18.1	18.0	17.8
Residual lignin (%)	17.5	17.3	16.8	16.8	15.6	15.0
pH	7.9	7.8	7.8	8.1	8.2	8.1
Residual NaOH (g·l-1)	0	0	0	0	0	0
Residual Na ₂ CO ₃ (g·1 ⁻¹)1	18.7	16.9	15.8	52.6	52.1	52.1
Weight (g·m ⁻²)	127	127	127	127	127	127
Thickness (µm)	262	260	253	252	252	255
Bulk density (g·cm ⁻³)	0.59	0.58	0.56	0.56	0.55	0.54
Tensile strength (kN·m ⁻¹)	2.28	2.28	2.30	2.35	2.40	2.47
Tensile index (Nm·g ⁻¹)	17.90	18.10	19.00	19.10	19.60	19.70
Breaking length (km)	2.31	2.35	2.46	2.48	2.54	2.56
Burst index (kPam ² ·g ⁻¹)	0.60	0.60	0.70	0.70	0.80	0.90
CMT ₃₀ (N)	85.00	87.00	90.00	102.00	105.00	114.00
SCT (kN·m ⁻¹)	1.00	1.00	1.00	1.10	1.20	1.30
Gurley (s)	1.20	1.30	1.30	1.60	1.70	1.70

The conditions and results of the laboratory scale cooking of disintegrated PB and OSB using the mildly alkaline sulphur-free technology for a preparation of semichemical pulp are shown in Tab. 4 and Tab. 5. Under the same batch conditions, the PB disintegrated waste was processed with up to 3.4 - 6.3 % lower yields compared to the OSB disintegrated waste yields. The yields of the PB disintegrated waste were higher up to 5 % compared to the cube-shaped PB samples, which may be related to the separation of a certain amount of glue due the disintegration and sorting. When cooking the PB disintegrated waste, a more selective degradation of lignin has occurred compared to the cube-shaped samples cooking, as evidenced by the lower residual lignin in semichemical pulp. The lower proportion of a non-cooked matter in the PB disintegrated waste also indicates the better impregnation when cooked.

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The treatment of the OSB waste by its disintegration had no effect on the yields achieved, on the residual lignin content and the content of the non-cooked matter in semichemical pulp obtained compared to the cube-shaped OSB waste. The waste OSB treatment by its disintegration resulted in only a small decrease in the yield, the residual lignin content and the non-cooked matter.

Extending the cooking time and increasing the content of alkali resulted in a deeper cooking, which is confirmed by the lower yields for PB and OSB disintegrated wastes as well as a slight decrease in residual lignin in semichemical pulp.

The high content of the non-cooked matter, which ranged from 13.9 - 21.1 % at yields 73.2 - 84.8 % for both the PB and OSB wasted treated by disintegration, indicates a poor waste impregnation, resp. an insufficient amount of chemicals in the cooking solution, especially NaOH, where a residual content in the leachate is zero after the cooking.

From the view of the mildly alkaline sulphur-free production of the semichemical pulp, the OSB waste appears to be a more advantageous material which achieved better strength parameters as CMT₃₀, SCT and burst index, which are decisive for the fluting.

From the evaluation of mechanical properties for the semichemical pulp prepared from the disintegrated PB and OSB waste on lab sheets 127 g·m⁻² (see Tab. 4 and Tab. 5) follows that only the sample A4/12 (OSB with a yield of 76.6 %, CMT_{30} =114 N and SCT = 1.32 kN·m⁻¹) meets the fluting criteria (**LWM - Light weight medium fluting**). Such recovery of wood agglomerates appears to be disadvantageous compared to paper recycled fibers.

It follows from the above that semichemical pulps from the disintegrated waste of PB and OSB prepared using the mildly alkaline sulphur-free technology showed low strength parameters. Therefore, the effect of the addition of OCC on the change of the strength parameters of laboratory prepared semichemical pulps was monitored. The change of strength parameters was monitored on samples with the best strength parameters, i.e., sample A4/6 and A4/12 with OCC addition of 20-60 %. Particularly, the parameters that are decisive for the production of fluting and liners, namely burst index, CMT_{30} and SCT were observed. Strength parameters were compared on lab sheets weighing 127 gm⁻².

The increase of the observed strength parameters of the semichemical pulp due to the addition of OCC is seen on Fig. 1- Fig. 3.



Fig. 1: Influence of the OCC addition to the Fig. 2: Influence of the OCC addition to the semichemical pulp sample on the burst index semichemical pulp sample on CMT_{30} (127gm⁻²) (127gm⁻²)



Fig.3:Influence of the OCC addition to the semichemical pulp sample on $SCT(127g m^{-2})$

Regarding the burst index and CMT_{30} , each 20% addition of OCC meant a 20% increase in these parameters. A slightly smaller increase was observed in the SCT value. This value increased from 1.3 (resp. 1.2 kN·m⁻¹) up to 1.8 kN·m⁻¹ for the semichemical pulp with the 60% OCC addition.

CONCLUSIONS

The evaluation of lab cooking of cube-shaped samples of waste PB and OSB shows, that: Under the same batch conditions of cube- shaped samples the PB waste processed up to 10 % lower yields compared to OSB.

- Extending the cooking time and increasing the supply of cooking chemicals has resulted in deeper overcooking.
- The high content of non-cooked matter indicates a poor impregnation of the cubes, which can be caused by the high content of cured adhesives among/inside the wood particles.
- Better strength parameters have been achieved in lab sheets made of OSB waste to compare to PB waste.
- The prepared semichemical pulp is not suitable for the production of the least quality liners, because the strength parameters were relatively low and did not correspond nor to 30 % of the required values.

The evaluation of lab cooking of disintegrated and sorted samples of waste PB and OSB (fraction 4-8 mm) shows, that:

- Under the same batch conditions, the PB disintegrated waste was processed up to 3.4 6.3 % lower yields of semichemical pulp compared to OSB disintegrated waste.
- The yields of semichemical pulp from PB disintegrated waste were higher by 5% compared to cube-shaped samples, which may be related to the separation of a certain amount of glue and sorting as well.
- Mechanical treatment of the OSB waste by its disintegration had no effect on yields achieved, the residual lignin content or the content of non-cooked matter compared to cubeshaped samples.
- Extending the cooking time and increasing the content of the cooking chemicals resulted in a deeper overcooking.
- Waste OSB material appears as a more advantageous material from the point of view of the
 production of semichemical pulp and its processing into fluting and liners.
- From the evaluation of the mechanical properties follows that only the sample prepared from OSB with a yield of 76.6 %, CMT₃₀=114 N and SCT=1.32 kN·m⁻¹ meets criteria for LWM - Light weight medium fluting.

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The evaluation of lab cooking of disintegrated and sorted samples of waste PB and OSB (fraction 4 - 8 mm) with an addition of OCC shows, that:

- Already the 40 % addition of OCC has contributed to the increase in strength parameters of semichemical pulp such that these were suitable for the production of "Recycled fluting medium", resp. "Brown Testliner 4".

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