

# Potential of pulp and paper secondary sludge as co-adhesive and formaldehyde scavenger for particleboard manufacturing

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**Abstract** This study investigated the potential of secondary sludge (SS) as urea–formaldehyde (UF) co-adhesive for particleboard manufacturing. Three proportions of SS from three conventional pulping processes were added in the formulation of particleboard manufacturing. A 3<sup>3</sup> factorial design was used. All panels were tested for thickness swell (TS), linear expansion (LE), internal bond strength (IB), flexural modulus of elasticity (MOE), flexural modulus of rupture (MOR) and formaldehyde emission. Results indicated that particleboards made with SS from thermomechanical pulp (TMP) and kraft pulp (Kraft) met the ANSI standards for LE, IB, MOE, and MOR (with 7 and 9 % UF). However, the TS of panels made with SS was higher than that of control panels and adding SS to the formulation affected negatively this property. Most of the properties studied in the particleboards made with SS from chemical–thermomechanical pulping (CTMP) process failed to meet the ANSI standards. The main advantage of using SS as co-adhesive is the reduction of formaldehyde

emission, in the best case here, about 50 %, with CTMP sludge added, of the particleboards.

## Verwendung von Sekundärschlamm aus der Zellstoffherstellung als Klebstoffzusatz und Formaldehydfänger bei der Spanplattenherstellung

**Zusammenfassung** In dieser Studie wird die mögliche Verwendung von Sekundärschlamm (SS) als Klebstoffzusatz von Harnstoffformaldehydharz (UF) bei der Spanplattenherstellung untersucht. Bei der Spanplattenherstellung wurden drei unterschiedliche Anteile an Sekundärschlamm aus drei konventionellen Aufschlussverfahren der Rezeptur beigegeben. Es wurde ein 3<sup>3</sup> Faktoren-Versuchsplan verwendet. Geprüft wurde die Dickenquellung (TS), die Längenausdehnung (LE), die Querkzugfestigkeit (IB), der Biege-Elastizitätsmodul (MOE), die Biegefestigkeit (MOR) und die Formaldehydabgabe der Platten. Die Ergebnisse zeigen, dass Spanplatten, die aus Sekundärschlamm von thermomechanischem Zellstoff (TMP) und Kraft-Zellstoff (Kraft) hergestellt wurden, die Anforderungen der ANSI Standards bezüglich Längenausdehnung, Querkzugfestigkeit, Biege-E-Modul und Biegefestigkeit (mit 7 und 9 % UF) erfüllten. Jedoch war die Dickenquellung der mit Sekundärschlamm hergestellten Platten höher als die der Kontrollplatten und die Zugabe von Sekundärschlamm zur Rezeptur wirkte sich negativ auf diese Eigenschaft aus. Bei mit Sekundärschlamm aus dem chemisch-thermomechanischem Aufschlussverfahren (CTMP) hergestellten Spanplatten erfüllten die meisten der untersuchten Eigenschaften die Anforderungen der ANSI Standards nicht. Der wesentliche Vorteil der Verwendung von Sekundärschlamm als Klebstoffzusatz ist die Reduzierung der Formaldehydabgabe der Spanplatten bei der Zugabe von CTMP-Schlamm, die im besten Fall bis zu 50 % betrug.

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## 1 Introduction

Wastewater treatment processes in pulp and paper mills generate large amounts of solid residues known as pulp and paper sludge. Sludge production accounted for about 4.8 % of mill production in 2006 (MDDEP 2009). The water treatment process typically includes primary treatment followed by secondary treatment (Smook 2002). Primary treatment removes suspended solids from wastes. The solid residue obtained after thickening is called primary sludge (PS). Waste waters from primary treatment go to secondary treatment, also called biological treatment, and the solid residue obtained after thickening is called secondary sludge (SS). The main organic components of SS are microbial extracellular polymeric substances (EPS), non-biodegraded materials, and microbial cells (Bitton 2005).

Currently, the most common sludge disposal methods are landfilling, incineration for power production, and land application for soil amendment (Mahmood and Elliott 2006; Smook 2002). However, current disposal alternatives suffer from shrinking space, public opposition, stricter regulatory pressures, and above all, poor economics (Mahmood and Elliott 2006; Smook 2002). Recent studies showed that sludge could be used for medium density fiberboard (MDF) and particleboard manufacturing with and without resins (Davis et al. 2003; Geng et al. 2006; Geng and Zhang 2007; Migneault et al. 2010, 2011b; Pervaiz and Sain 2011; Taramian et al. 2007). The use of sludge was beneficial for improving the internal bond strength mainly due to the bonding potential of SS.

Migneault et al. (2011b) reported that replacing a proportion of the UF resin by SS in MDF led to a reduction in formaldehyde emissions by up to 68 % over control panels without compromising the internal bond strength. Thus, in addition to improving adhesion or reducing resin content, adding SS in the fiberboard manufacturing might have beneficial environmental effects and important practical implications: SS could be applied as a HCHO scavenger for UF-bonded fiberboards. However, this study warned that although SS has significant bonding properties, more effort is needed to use it as a co-binder with synthetic resins due to several matters including its higher pH and lower buffering capacity compared to wood fibers. In addition, the resin gel time is considerably reduced when SS is added.

In recent years, interest in bio-based adhesives has increased due to increasingly stringent government regulations in the use of synthetic adhesives. Bio-based adhesives are derived from natural materials such as proteins, carbohydrates and lignin (Pizzi 2006; Rowell 2005). Secondary sludge from paper mill contains some of these substances, and already showed good bonding ability (Geng et al. 2007; Zerhouni et al. 2012) Considering the

bonding ability of SS, and the substantial amount of resin used by the wood composites industry, the use of SS as co-adhesive with urea–formaldehyde resin (UF) is an interesting option to recycle this residue in particleboard manufacturing.

Urea–formaldehyde (UF) is the main adhesive used in particleboard manufacturing. Its advantages include low cost, ease of use under a wide variety of curing conditions, low cure temperatures, water solubility and lack of color. The major disadvantage associated with UF as compared to other thermosetting wood adhesives is the lack of resistance to moist conditions, especially in combination with heat. These conditions lead to a reversal of the bond-forming reactions and the release of formaldehyde. For this reason, UF resins are usually used for the manufacture of products intended for interior use only. However, even when used for interior purposes, the slow release of formaldehyde (a suspected carcinogen) from products bonded with UF adhesives is a major concern.

In a previous manuscript (Xing et al. 2012), data on the physical and mechanical properties of panels made with TMP and Kraft SS sludges were reported. The same data, along with additional data on CTMP SS sludge, is used also in this study for statistical data analysis purposes, along with other data on chemical compositions of sludges and on formaldehyde emissions of panels. Thus, the main objective of this study was to examine the potential of SS from three pulping processes as co-adhesive in particleboard manufacturing. Specific objectives were: (1) to characterize the chemical properties of SS from three pulping processes, (2) to evaluate the impacts of UF content and SS proportion from three different pulping processes on selected physical and mechanical properties of particleboards, (3) to investigate the potential of SS as co-adhesive to reduce formaldehyde (HCHO) emissions.

## 2 Materials and methods

### 2.1 Secondary sludge collection, drying and grinding

Thermomechanical pulp (TMP) SS was collected from the Stadacona White Birch Pulp and Paper Mill in Québec City (Quebec, Canada). Kraft pulp (Kraft) SS was collected from the SFK Pulp Fund Commercial Pulp Mill in Saint-Félicien (Quebec, Canada). Chemical–thermomechanical pulp (CTMP) SS was collected from Tembec Matane Inc. (Matane, Canada). Samples were refrigerated at 4 °C, squeezed and dried at 60 °C for 1 week to avoid protein deterioration. The dried SS samples were ground in a roller mill. The milled material was further screened with a 30 mesh sieve (600 µm) and the fraction was collected. The collected material was used to manufacture the

particleboard. A small part of this collected material was screened again. The fraction between 40 mesh sieve and 60 mesh sieve was placed in airtight container to stabilize the moisture content and then used for chemical analysis. The adhesive used was UF (UL 232, Arclin Canada, Sainte-Thérèse, Quebec, Canada).

## 2.2 Secondary sludge and particle characterization

The chemical composition of secondary sludge from three pulping processes is presented in Table 1. Ash content was determined by combustion in a muffle furnace at 525 °C according to TAPPI-211 om-93 (Tappi standard 1993a). Total extractive content was determined by successive extractions with an organic solvent mixture (ethanol/toluene) followed by hot water extraction according to TAPPI T 204 cm-97 (Tappi standard 1997) and T 207 om-93 (Tappi standard 1993b). Cellulose content was determined by Kürschner and Hoffer's nitric acid method. Pentosan content was obtained according to TAPPI T 223-84 (Tappi standard 1984). Total lignin content was determined by the Klason method according to TAPPI T 222 om-98 (Tappi standard 1998, acid-insoluble lignin) and by absorption spectroscopy at 205 nm according to TAPPI useful method um-250 (Tappi useful method 1985, acid-soluble lignin). Nitrogen content was determined using a Perkin Elmer (Waltham, MA) 2410 Series II nitrogen analyzer. Three repetitions were conducted for chemical analysis. Because the CTMP SS contains high soluble inorganic materials and high protein, inorganics contribute to an overestimation of Klason lignin and holocellulose, for samples with high protein content, a fraction of the protein also contaminates the Klason lignin and the holocellulose. Then cellulose content and lignin content had to be corrected according to the new analytical method (Rabemanolontsoa et al. 2011).

It is worthy to note that the methods used to determine sludge chemical properties are designed for wood chemical analysis and might not be appropriate for sludge. In the absence of specific standard methods for sludge chemical analysis, the use of the wood standard methods is a reasonable compromise as suggested in previous

reports (Migneault et al. 2010, 2011a, b; Pervaiz and Sain 2011).

Buffering capacity and pH were measured according to the method proposed by Johns and Niazi (1980) with some modifications due to the SS being different from wood. An aqueous extract was prepared by refluxing 25 g of dry furnish in 250 mL distilled water for 20 min. Extract solution was then filtered through centrifugation, since the SS contains a large amount of soluble materials and filter paper was rapidly clogged. Extraction solution was cooled at room temperature before titrating. Two extract solutions were prepared for each furnish type. The extract (50 mL) was titrated to a pH of 3 using 0.25-N H<sub>2</sub>SO<sub>4</sub> solution for alkaline buffering capacity or to a pH of 8 using 0.25-N NaOH solution for acid buffering capacity. Two titrations were conducted for each titration solution. Thus, the initial pH value for each sample is the average of eight measurements, while each buffering capacity value (mmol/100 g oven dried sample) is the mean of four measurements.

## 2.3 Particleboard manufacturing

The particleboards were manufactured in the Centre de Recherche sur le Bois (CRB, université Laval, Québec, Canada). The wood particles used [a mixture of spruce (*Picea*), fir (*Abies*), and pine (*Pinus*)] were obtained from TAFISA, a particleboard mill in Lac-Mégantic, Québec, Canada. The size of particles ranged from 1 to 5 mm and the moisture content ranged from 2 to 4 %.

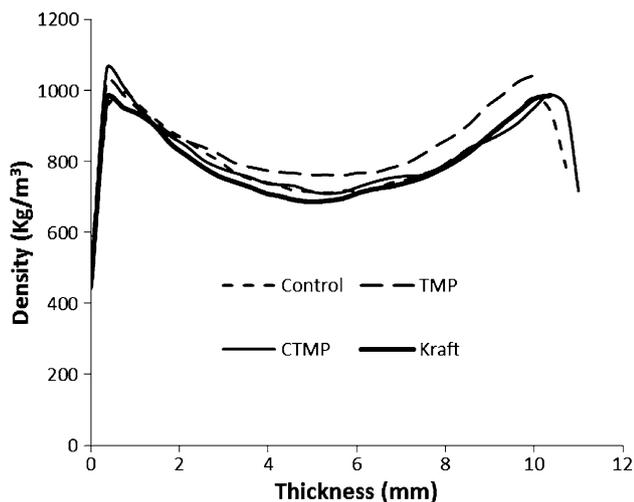
Particleboards were processed according to a 3<sup>3</sup> factorial design, where the factors were the content of UF resin (5, 7, and 9 % dry weight of resin per dry weight of wood particles), the types of secondary sludge (SS: TMP, CTMP, and Kraft) and SS content (75, 100, and 125 % dry weight of SS per dry weight of resin). Three particleboard panels were made for each experimental condition (three repetitions). Three control panels were also made for each UF's content. A total of 90 panels were produced. The panels target density was 750 kg/m<sup>3</sup>, pressing temperature was 210 °C, and pressing cycle was 6.0 min (including closing and opening time). The pressing schedule was optimized to

**Table 1** Chemical composition of secondary sludge (SS) from the three pulping processes  
**Tab. 1** Chemische Zusammensetzung des Sekundärschlammes (SS) der drei Aufschlussverfahren

Pulping process	Ash (%)	Extractives (%)	Cellulose (%)	Lignin (%)	Pentosan (%)	Nitrogen (%)
TMP <sup>a</sup> (Stadacona)	12.0 (0.1)	21.5 (0.6)	19.7 (0.2)	50.2 (0.5)	3.0 (0.1)	7.7 (0.0)
CTMP (Tembec)	31.2 (0.0)	43.1 (2.1)	8.9 (0.1)	23.2 (0.2)	2.2 (0.0)	4.7 (0.1)
Kraft <sup>a</sup> (SFK)	41.3 (1.4)	7.9 (0.0)	18.9 (0.4)	36.4 (1.0)	3.4 (0.0)	1.3 (0.0)

The numbers in parentheses are standard deviation values

Data for TMP<sup>a</sup> and Kraft<sup>a</sup> came from Migneault et al. (2011a, b)



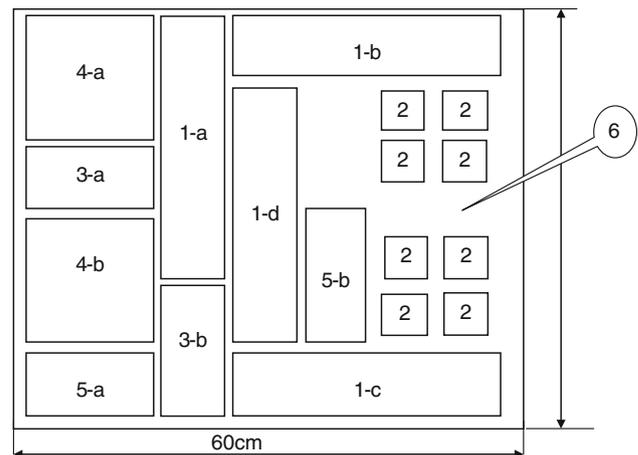
**Fig. 1** Examples of density profiles of particleboards made from SPF particles with 7 % UF and 100 % SS as co-adhesive from three pulping processes (each curve is an average for eight samples from the same panel)

**Abb. 1** Dichteprofil von Spanplatten, die aus SPF-Spänen und 7 % UF und 100 % SS von drei Ausschussverfahren als Klebstoffzusatz hergestellt wurden (jede Kurve entspricht dem Mittelwert von 8 Prüfkörpern derselben Platte)

obtain similar density profiles for all panels (Fig. 1). The target thickness was 11 mm. For different UF contents, 0.5 % dry weight of emulsion wax (EW58A, Hexion Canada, Québec, Canada) per dry weight of particles and 0.20 % dry weight of ammonium chloride (as a catalyst) per dry weight of resin were mixed and diluted to the appropriate consistency to reduce viscosity. First, dry SS was added to the particles in a conventional rotary drum blender mounted with a spray nozzle. Then, the mixture of UF resin, water, wax, and ammonium chloride was added to the blender through the spray nozzle. Total furnish MC was 12 %. Mats were hand-formed in a 500 × 600 mm mold and pressed in a Dieffenbacher hydraulic press with a 1,000 × 1,000 mm plate. This two-step procedure was adopted since preliminary tests showed that it is not possible to mix UF and SS prior to particle resination, since immediate reaction between the two resulted in resin blend coagulation (Xing et al. 2012). Hence the UF–SS reaction anticipated in this case may take place in situ during pressing.

#### 2.4 Panel testing

All panels were conditioned at  $20 \pm 3$  °C and 65 % relative humidity (RH) until equilibrium moisture content was reached. The panels were cut to extract test samples according to the drawing shown in Fig. 2. The internal bond (IB), flexural modulus of rupture (MOR) and modulus of elasticity (MOE), thickness swelling (TS), and linear



**Fig. 2** Drawing for the panel cutting for test samples extraction: 1 four 314 × 75 mm specimens for bending tests, 2 eight 50 × 50 mm specimens for the internal bond and density profile, 3 two 150 × 75 mm specimens for Janka hardness and formaldehyde emission, 4 two 150 × 150 mm specimens for the thickness swelling and formaldehyde emission, 5 two 150 × 75 mm specimens for the linear expansion, 6 thermocouple trace avoided in specimen sampling

**Abb. 2** Schnittplan zur Herstellung der Prüfkörper: 1 Vier 314 × 75 mm Prüfkörper für die Biegeprüfung, 2 Acht 50 × 50 mm Prüfkörper für die Querkzugfestigkeit und das Dichteprofil, 3 Zwei 150 × 75 mm Prüfkörper für die Janka Härte und die Formaldehydabgabe, 4 Zwei 150 × 150 mm Prüfkörper für die Dickenquellung und die Formaldehydabgabe, 5 Zwei 150 × 75 mm Prüfkörper für die Längenausdehnung, 6 Bei der Probenahme wurde sichergestellt, dass Bohrungen für die Thermoelemente ausgeschlossen wurden

expansion (LE) were measured according to ASTM D 1037-2006a (ASTM D 1037 2006) methods and were compared with the ANSI A 208.1-2009 standard (ANSI 2009). The formaldehyde emission level was measured by means of the desiccator method according to ASTM D 5582-00 (2000) with the following modifications: two samples of 75 × 150 mm and two samples of 150 × 150 mm were implemented and no wax was applied to sample edges. Density profiles of each IB specimen after sanding were measured with a QMS density profile system, version 1.25.

#### 2.5 Statistical data analysis

A statistical data analysis was performed by means of a SAS package version 9.2 (SAS Institute 2007). A factorial analysis of variance was used to evaluate the effects of UF proportion (UFP), SS source (SSS) and SS proportion (SSP) within UF resin. All factors were considered fixed. The effects of the studied factors on the panels' physical and mechanical properties were tested at 95 % confidence level using a mixed procedure (SAS Institute 2007). Means

were compared using the Waller–Duncan multiple comparison test.

During the hot pressing, the interaction among heat, moisture and pressure gives rise to a non-uniform deformation of the wood particle mat, which results in an uneven density distribution across the thickness of the board. Typically, this density profile resembles a ‘U-shape’, with the peak density near the board surfaces, and the lowest density in the core region (Wong et al. 1999). The presence of this vertical density gradient has been reported to result in higher bending strength, but lower internal bond and interlaminar shear (Kelly 1977). So in order to remove the variation in panel physical and, especially, mechanical properties caused by sample density variations and the presence of vertical density gradient in individual sample, all properties were adjusted according to Eqs. (1) and (2):

$$\text{Adjusted physical property} = \frac{\text{measured property}}{\text{target density}} \times \text{average density} \quad (1)$$

$$\text{Adjusted mechanical property} = \frac{\text{measured property}}{\text{average density}} \times \text{target density} \quad (2)$$

where the target density was 750 kg/m<sup>3</sup>.

The average density is the sample density. For IB, the average density represents the sample core density obtained from X-ray density profiles. Since the density of broken (core) layer strongly affects the IB, the tested IB should be adjusted to the same core density level before statistical analysis (Xing et al. 2006).

### 3 Results and discussion

#### 3.1 Secondary sludge properties

The chemical composition of SS from the three mills is presented in Table 1. Results indicate high differences in all chemical properties of SS from the three different pulping processes. These differences are due to several factors including the chemical products used for pulping, bleaching, and other operations in each process. Differences in primary and secondary treatments of sludge also can affect

the chemical composition of the SS. Ash in SS comes from non-woody materials rejected in wastewaters at any stage of pulp and paper processing, such as dirt from chips cleaning, papermaking chemicals, papermaking fillers, or rejected inert solids (Ochoa de Alda 2008; Smook 2002). The ash content was highest in the Kraft SS followed by the CTMP and TMP SS, respectively. The Kraft process uses high quantities of chemicals for pulping which explains in part its highest ash content in the SS. In the CTMP process, chemicals are used but to a much lesser extent than the Kraft process explaining the lower ash content in its SS. The TMP SS sludge has the lowest ash content due to the fact that no chemicals are used in the pulping process.

The Kraft SS has the lowest extractive content compared to TMP and CTMP SS (Table 1). The extractive content was surprisingly high in the CTMP SS. The lignin content was also relatively high in SS, particularly in TMP SS. This could be explained by the fact that lignin is found not only in fiber cell walls but also in other chemical by-products such as polyphenols (Migneault et al. 2010, 2011a, b; Zerhouni et al. 2012). Lignin contents may also have been interfered with by phenol-like molecules not found in wood, resulting in a total material slightly higher than 100 % (Table 1).

Carbohydrates content is low in SS. This result is in agreement with previous reports (Geng et al. 2007; Pervaiz and Sain 2011; Smook 2002; Zerhouni et al. 2012) and is due to the fact that SS contains few wood fibers. SS has high nitrogen content, inferring the presence of proteins. Protein-rich SS is expected to reduce HCHO emissions, because protein molecules contain many functional groups that may react with HCHO, such as amines and amides (Dutkiewicz 1984; Lorenz et al. 1999; Wescott et al. 2006). Protein-rich SS is also expected to have a positive effect on IB strength, as shown by the use of proteins in wood adhesive formulations (Pizzi and Mittal 2003; Rowell 2005) due to its higher nitrogen content.

Buffering capacity and pH of the SS and particles used in panel formulations are presented in Table 2. Acidity from the furnish is required for UF resin to polymerize and build a strong cross-linked network (Rowell 2005). A pH of about 4–5 or lower is recommended to obtain a reasonable pressing time (Maloney 1993; Rowell 2005). The

**Table 2** Buffering capacity and pH of SS and wood particles

**Tab. 2** Pufferkapazität und pH-Wert des Sekundärschlammes und der Holzspäne

	Wood particles	TMP SS	CTMP SS	Kraft SS
pH	4.50	6.10	9.40	6.76
Acid buffering capacity (mmol/100 g oven dried sample)	0.50	3.16	–	0.32
Alkaline buffering capacity (mmol/100 g oven dried sample)	0.34	4.83	18.22	0.56

**Table 3** Analysis of variance of the effect of urea–formaldehyde proportion (UFP), secondary sludge source (SSS) and secondary sludge proportion (SSP) on selected physical and mechanical properties of particleboards**Tab. 3** Ergebnisse der Varianzanalyse bezüglich des Einflusses des Harnstoffformaldehydgehalts (UFP), der Art des Sekundärschlamms (SSS) und des Sekundärschlammanteils (SSP) auf die untersuchten physikalischen und mechanischen Eigenschaften der Spanplatten

Fixed effects	DF	Physical properties			Mechanical properties		
		TS (24 h)	LE	FE	IB	MOE	MOR
UFP	2	128.6**	0.6 ns	4.76*	200.1**	93.7**	95.7**
SSS	2	250.4**	52.5**	67.26**	104.1**	79.0**	86.0**
UFP × SSS	4	16.5**	–	–	59.2**	4.8**	13.0**
SSP	3	302.1**	6.2**	36.45**	72.5**	30.0**	63.2**
UFP × SSP	6	2.7*	–	–	6.6**	10.8**	4.7**
SSS × SSP	6	30.3**	6.9**	8.63**	13.0**	9.2**	10.0**
UFP × SSS × SSP	12	5.3**	–	–	14.8**	2.6**	3.5**

ns Non-significant at the 95 % probability level

\*\* Significant at the 99 % probability level

\* Significant at the 95 % probability level

pH of particles used in this study (4.5) falls within this acceptable range while that of the SS from the pulping processes was well above this range (6.10–9.40). Of the SS sources, TMP SS is the best candidate in terms of pH for panel manufacturing, CTMP SS is the worst. A resin catalyst (such as ammonium chloride) can decrease the furnish pH, but a low alkaline buffering capacity is preferable. In terms of buffering capacity, Kraft SS is the best candidate for panel manufacturing and CTMP SS the worst.

### 3.2 Variation of the panel physical and mechanical properties

The results of the analysis of variance on the effects of UFP, SSS, and SSP on the physical and mechanical properties are summarized in Table 3. It is worthy to note that SS source and proportion have highly significant effects ( $P < 0.001$ ) on all physical and mechanical properties. The effect of UFP is highly significant ( $P < 0.001$ ) on thickness swelling and all mechanical properties (Table 3), and is significant on formaldehyde emission ( $P = 0.0143$ ). However, its effect on linear expansion was not significant.

#### 3.2.1 Thickness swell (TS)

The variation of thickness swelling with SS source, UF proportion and SS proportion along with Waller–Duncan multiple comparisons is shown in Table 4. According to ANSI A208.1 standard (2009), maximum thickness swelling values for home decking and load bearing particleboards are 8 and 14 %, respectively. All panels had higher thickness swelling values than those allowed by the standards. As expected, increasing UFP from 5 to 7 % tends to decrease the TS after 24 h water immersion. This result is

expected and in good agreement with previous findings (Beech 1975; Halligan 1970; Lehmann 1974, 1978). However, the TS of panels made with 9 % UF do not follow a particular trend, in some cases it was comparable to that of panels made from 7 % UF and in other cases it is higher or even lower depending on the SS origin. These trends of variation explain the significant interaction UFP × SSS (Table 3).

The source of SS also showed significant effect on the TS after 24 h water immersion. In general, using TMP SS as co-adhesive showed lower TS values than CTMP and Kraft SS. Chemical composition of the SS could be a plausible explanation. The TMP SS showed higher lignin content than CTMP and Kraft SS (Table 1). Lignin is known to be hydrophobic and its beneficial effect on the dimensional stability of panels is shown by previous researchers (Westin et al. 2001). The panels made with CTMP SS swelled more than those made with TMP and Kraft SS. This result could also be explained by the chemical composition of the CTMP SS which showed the lowest lignin content (23.2 %) and the highest extractives content (43.1 %). Extractives are undesirable for panel manufacturing. Because they may evaporate during hot-pressing and create delamination and decrease resin crosslinking (Maloney 1993). Also this could be due to the high pH and alkalinity of the CTMP SS (Table 2) which may hinder UF reticulation, leading to higher TS.

The SS proportion within the UF resin also showed highly significant effect on the TS. Increasing SSP led to an important increase in the thickness swelling in the case of TMP and CTMP SS. However, for Kraft SS, at any UFP, average TS was not significantly different (Table 4). This difference in TS response to SSP explains the significant interactions SSS × SSP and UFP × SSS × SSP (Table 3).

**Table 4** Panel Physical properties (standard deviation in parentheses) made with SS from three pulping processes (the case where SS/UF = 0 corresponds to the values of the control panels)**Tab. 4** Physikalische Eigenschaften der Platten (Standardabweichung in Klammern), die mit Sekundärschlamm aus den drei Aufschlussverfahren hergestellt wurden (SS/UF = 0 entspricht den Werten der Kontrollplatten)

Pulping process	UF (%)	SS/UF (%)	TS (24 h) (%)	Waller-Duncan Test	LE (%)	Waller-Duncan Test
TMP <sup>a</sup>	5	0	26.31 (0.99)	f	0.21 (0.02)	a
		75	42.10 (1.71)	cd	0.18 (0.03)	a
		100	50.60 (2.40)	b	0.18 (0.02)	a
		125	63.86 (4.27)	a	0.22 (0.03)	a
	7	0	14.10 (1.31)	h	0.19 (0.02)	a
		75	20.43 (3.27)	g	0.21 (0.04)	a
		100	42.99 (4.27)	cd	0.20 (0.02)	a
		125	36.82 (2.98)	e	0.19 (0.02)	a
	9	0	16.94 (1.24)	g	0.19 (0.03)	a
		75	40.40 (5.13)	cd	0.19 (0.02)	a
		100	40.17 (3.90)	de	0.22 (0.04)	a
		125	43.80 (5.00)	c	0.19 (0.02)	a
CTMP	5	0	21.63 (0.99)	f	0.21 (0.02)	de
		75	59.69 (4.41)	e	0.26 (0.03)	abc
		100	68.24 (3.74)	cd	0.24 (0.03)	bcd
		125	74.97 (3.88)	a	0.25 (0.03)	bcd
	7	0	14.10 (1.31)	g	0.19 (0.02)	e
		75	58.67 (2.73)	e	0.22 (0.03)	cde
		100	69.12 (5.51)	bc	0.25 (0.04)	bcd
		125	64.43 (4.37)	d	0.26 (0.03)	abc
	9	0	16.94 (1.24)	g	0.19 (0.13)	e
		75	64.34 (6.88)	d	0.24 (0.06)	cd
		100	73.09 (4.70)	ab	0.28 (0.04)	ab
		125	72.37 (5.98)	abc	0.29 (0.04)	a
Kraft <sup>a</sup>	5	0	26.31 (0.99)	d	0.21 (0.02)	ab
		75	60.61 (5.36)	a	0.20 (0.02)	ab
		100	59.31 (2.52)	a	0.18 (0.03)	b
		125	57.20 (5.97)	a	0.18 (0.02)	ab
	7	0	14.10 (1.31)	e	0.19 (0.02)	ab
		75	32.84 (5.73)	c	0.18 (0.02)	ab
		100	38.33 (3.89)	b	0.19 (0.02)	ab
		125	37.46 (3.85)	b	0.21 (0.02)	a
	9	0	16.94 (1.24)	e	0.19 (0.03)	ab
		75	26.52 (5.11)	d	0.20 (0.01)	ab
		100	29.84 (5.46)	cd	0.19 (0.02)	ab
		125	29.92 (3.71)	cd	0.18 (0.01)	ab

Means with the same letter are not significantly different from each other at the 5 % probability level compared to the same pulping process According to ANSI A 208.1-2009, for M-2 particleboard grade for interior species LE <0.40 %, with no special requirement for TS

<sup>a</sup> Data for TMP and Kraft are from Xing et al. 2012

Using SS from the three pulping processes as co-adhesive led to a very important increase in the panel TS compared to control panels (Table 4). This result is in good agreement with recent reports (Migneault et al. 2010, 2011b). The increase in thickness swell when using SS is

expected since most of protein based adhesives also suffer from poor dimensional stability (Pizzi 1989; Rowell 2005).

Considering the fact that thickness swell is among the most important properties in particleboard use for indoor applications, the use of SS as co-adhesive in its actual form

is not a viable option. Chemical modification or the addition of important hydrophobic components such as wax in the chemical mix is necessary. Further investigations are needed.

### 3.2.2 Linear expansion (LE)

The LE values are presented in Table 4. The average LE values ranged from 0.18 to 0.29 which is well below 0.40, the maximum value required by the M-2 particleboard grade for interior use specified by the ANSI A 208.1-2009.

The effect of UFP on LE was not significant while the effects of the SS source and proportion were highly significant (Table 3). The highest LE values were obtained for the panels where CTMP SS is used as co-adhesive (Table 4). Panels made with TMP and Kraft SS as co-adhesive led to similar LE values. The higher LE values with CTMP SS could be explained by the highest extractives content, especially soluble extractives content. When the RH increased from 50 to 80 %, the samples from CTMP SS panels absorbed much more water than those from TMP SS and Kraft SS panels, respectively. Panels made with TMP and Kraft SS as co-adhesive were not statistically different than control panels. These results suggest that LE is not negatively affected by the use of SS as co-adhesive from any of the three pulping process.

### 3.2.3 Internal bond strength (IB)

The IB results are shown in Table 5. As expected, UFP showed highly significant effect on IB (Table 3). Increasing UFP generally improves the IB (Table 5). The Waller-Duncan multiple comparisons showed that the difference between the three levels of UFP was highly significant (Table 5). Using SS as co-adhesive from CTMP process led to a significant decrease of the IB compared to control panels, but using SS as co-adhesive from TMP and Kraft processes led to a significant increase of the IB in some cases (Table 3). The interaction UFP  $\times$  SSS was also highly significant due to the fact that at a constant UFP, the effect of SS on IB varied depending on its source.

Panels made with CTMP SS led to the lowest IB values compared to those made with TMP and Kraft SS. This could be due to the high pH and alkalinity of the CTMP SS (Table 2) which may hinder UF reticulation. Higher SS proportion generally led to a decrease in IB (Table 5). However, no particular pattern of variation of IB with SS proportion among the three SS types explaining thus the significant interaction SSS  $\times$  SSP. The triple interaction UFP  $\times$  SSS  $\times$  SSP on IB was also highly significant. This effect could be explained by the experimental errors and the high level of variation of the IB response observed in this study (Table 4).

The IB of panels made with TMP SS as co-adhesive met the requirements of M-2 particleboard grade for interior use (0.40 MPa) according to ANSI A 208.1-2009 (Table 5). However, at 5 % UF, IB values for panels with TMP SS as co-adhesive were significantly lower than those of control panels. This result is in good agreement with previous reports (Geng et al. 2007; Migneault et al. 2010; Taramian et al. 2007). At 7 % UF combined with 75 % SS, the IB was improved greatly over that of control panels. When SS proportion increased to 100 and 125 %, the IB fell significantly compared to that of control panels. For panels with 9 % UF and 75 and 100 % SS, IB was significantly below that of control panels, but with 125 % SS, IB was equal to that of control panels. IB did not decrease linearly with added SS; instead, it showed a 'U-shaped' profile. This result departs from previous reports (Geng and Zhang 2007; Migneault et al. 2010; Taramian et al. 2007), they didn't find the increase trend of IB after the decrease. Although the mechanism is not fully understood, this discrepancy could be attributed to an optimal stoichiometric UF-SS ratio giving best properties due to a chemical crosslinking between UF and SS.

Most IB values of the panels made with Kraft SS as co-adhesive (Table 5) met the requirements of M-2 particleboard grade for interior use, except for 5 % UF with 75 and 100 % SS content. With 5 % UF content, the addition of 125 % SS had no significant effect on IB compared to control panels. With 7 % UF and 75 % SS, IB improved significantly compared to that of control panels, but IB decreased with increasing SS proportion. The same result was found with 9 % UF. Owing to the highest ash content of Kraft SS (48.1 %, Table 1), the negative effect of SS on IB increased rapidly. This result is in good agreement with recent reports (Migneault et al. 2010, 2011a).

For the panels made with CTMP SS as co-adhesive (Table 5), only one-third of the panels met the requirements of M-2 particleboard grade for interior use in terms of IB (0.40 MPa). For the three UF proportions, the use of SS as co-adhesive significantly decreased the IB. This decrease is linear. This phenomenon was attributed to the highest content of extractives of CTMP SS (56.7 %) and higher ash content (31.2 %). Extractives are undesirable for panel manufacturing, since they may evaporate and degrade during hot-pressing, create delamination and decrease resin crosslinking (Maloney 1993).

### 3.2.4 Bending modulus of elasticity (MOE)

For all panels and formulations, increasing the UFP generally improved the MOE (Table 5). The use of SS from the three pulping processes as co-adhesive has a significant effect on the MOE but this effect is not always negative (Table 5). Depending on the SS source and the UFP

**Table 5** Panel mechanical properties (standard deviation in parentheses) made with SS from three pulping processes (the case where SS/UF = 0 corresponds to the values of the controls panels)

**Tab. 5** Mechanische Eigenschaften der Platten (Standardabweichung in Klammern), die mit Sekundärschlamm aus den drei Aufschlussverfahren hergestellt wurden (SS/UF = 0 entspricht den Werten der Kontrollplatten)

Pulping process	UF (%)	SS/UF (%)	IB (kPa)	Waller–Duncan Test	MOE (GPa)	Waller–Duncan Test	MOR (MPa)	Waller–Duncan Test
TMP <sup>a</sup>	5	0	499 (45)	f	3.02 (0.17)	ef	12.98 (1.43)	ghi
		75	457 (44)	gh	2.95 (0.15)	f	11.98 (2.02)	i
		100	429 (37)	h	2.99 (0.31)	ef	11.99 (1.97)	i
		125	442 (37)	h	3.03 (0.18)	ef	12.09 (1.80)	hi
	7	0	688 (153)	bc	3.12 (0.27)	de	17.01 (2.44)	ab
		75	771 (79)	a	3.64 (0.29)	a	17.50 (2.42)	a
		100	486 (48)	fg	3.13 (0.20)	de	14.17 (1.02)	efg
		125	576 (59)	e	3.20 (0.19)	d	13.51 (2.47)	fgh
	9	0	715 (62)	b	3.53 (0.20)	a	16.22 (1.60)	abc
		75	678 (45)	c	3.53 (0.12)	ab	15.76 (1.92)	bcd
		100	636 (45)	d	3.37 (0.19)	bc	15.24 (1.60)	cde
		125	692 (36)	bc	3.26 (0.20)	cd	14.55 (2.20)	def
CTMP	5	0	499 (45)	b	3.02 (0.17)	bc	12.98 (1.43)	b
		75	481 (43)	bc	2.77 (0.19)	de	12.02 (1.54)	bc
		100	451 (40)	c	2.62 (0.23)	e	10.22 (1.22)	de
		125	381 (68)	de	2.66 (0.34)	e	10.13 (2.13)	de
	7	0	688 (153)	a	3.12 (0.27)	b	17.01 (2.44)	a
		75	476 (58)	bc	3.00 (0.23)	bc	12.25 (1.74)	bc
		100	398 (61)	d	2.92 (0.38)	cd	11.16 (1.90)	cd
		125	375 (80)	de	2.76 (0.23)	de	10.20 (2.02)	de
	9	0	715 (62)	a	3.53 (0.20)	a	16.22 (1.60)	a
		75	343 (74)	f	2.93 (0.38)	bcd	11.08 (1.89)	cd
		100	283 (70)	g	2.68 (0.23)	e	9.58 (1.84)	e
		125	325 (82)	f	2.74 (0.23)	de	9.96 (1.96)	de
Kraft <sup>a</sup>	5	0	499 (45)	g	3.02 (0.17)	de	12.98 (1.43)	efg
		75	353 (28)	h	2.88 (0.14)	e	11.73 (1.22)	g
		100	271 (54)	i	2.64 (0.18)	f	10.27 (1.01)	h
		125	474 (46)	g	2.97 (0.25)	de	12.39 (1.56)	fg
	7	0	688 (153)	cd	3.12 (0.27)	cd	17.01 (2.44)	ab
		75	746 (50)	b	3.39 (0.21)	ab	15.92 (1.95)	bc
		100	663 (89)	d	3.31 (0.19)	b	15.36 (1.41)	cd
		125	572 (59)	f	3.08 (0.26)	d	13.60 (2.17)	ef
	9	0	715 (62)	bc	3.53 (0.20)	a	16.23 (1.60)	bc
		75	838 (100)	a	3.51 (0.23)	a	17.89 (1.03)	a
		100	651 (76)	de	3.11 (0.21)	cd	14.20 (1.53)	de
		125	614 (92)	e	3.27 (0.35)	bc	15.16 (3.26)	cd

Means with the same letter are not significantly different from each other at the 5 % probability level compared to the same pulping process According to ANSI A 208.1-2009, for M-2 particleboard grade for interior use IB >400 kPa, MOE >2.0 GPa, MOR >13 MPa

<sup>a</sup> Data for TMP and Kraft are from Xing et al. 2012

proportion, the MOE either remained constant, increased or decreased. This variability of the response explains the significant interactions obtained (Table 3). For example, the use of TMP SS as co-adhesive either improved or maintained constant the MOE compared to that of control

panels at any UF proportion. Increasing TMP SS proportion is not usually associated with a MOE improvement. On the other hand, the use of CTMP SS as co-adhesive decreased the MOE at any UF proportion (Table 5). Increasing the SS proportion led to a further decrease in the

MOE. The high inorganic material content of CTMP SS (up to 74.3 %) severely affects the MOE. For particleboards made with Kraft SS, the MOE of particleboards made with 5 % UF did not vary with SS proportion (Table 5). At 7 % UF, the MOE increased slightly with increasing SS proportion. However, at 9 % UF, the MOE slightly decreased with higher SS content. The results indicated that the impact of Kraft SS on MOE was not so serious.

Compared to TS and IB, the lesser effect of SS on the MOE could be explained by the fact that bending properties are less affected by adhesive performance (Maloney 1993). In fact, despite the significant differences between the various experimental conditions, all panels met the requirements of M-2 particleboard grade for interior use according to ANSI A 208.1-2009 (2.0 GPa). Compared to MOE of control panels, TMP panels were superior, Kraft panels were either similar or superior and CTMP panels were lower.

### 3.2.5 Bending modulus of rupture (MOR)

Increasing the UFP generally improved the MOR but the use of SS from the three pulping processes as co-adhesive slightly decreased the MOR (Table 5). However, the magnitude of reduction of MOR due to SS addition was not the same for the different UF proportion and different pulping processes. These differences explain the highly significant effects of the interactions reported in Table 3.

At 5 % UF, the minimum requirements of M-2 particleboard grade for interior use (13 MPa) were not met for almost all SS proportions from the three pulping processes except for the control panels (Table 5). At higher UF proportions, all particleboards with TMP and Kraft SS as co-adhesive met the standard requirements of M-2 particleboard grade for interior use (ANSI A 208.1-2009). However, none of the CTMP particleboards made with SS as co-adhesive (Table 5) met this requirement.

The use of SS as co-adhesive had a negative effect on MOR. In most cases, the MOR of panels made with SS as co-adhesives are lower than that of control panels (Table 5). This effect is highly significant (Table 3). For all particleboards, a general decrease tendency of the MOR with increasing SS proportion is observed (Table 5). This decrease could be attributed to weak adhesion between the SS and wood particles, due to the presence of inorganic materials in the sludge, such as kaolin clay and calcium carbonate.

Increasing the SS proportion decreases the MOR to reach a plateau between 100 and 125 %. The difference between MOR of panels made with TMP and Kraft SS was not significant. However, the MOR of CTMP panels was different from that of Kraft and TMP panels (<0.0001).

CTMP panels showed the lowest MOR values because the SS had high extractives content (43.1 %), high ash content (31.2 %), high pH value (9.40) and high alkaline buffering capacity (18.22 mmol/100 g oven dried sample).

### 3.2.6 Formaldehyde emission (FE)

The effects of UF resin proportion, SS source and SS proportion from three pulping processes on FE are shown in Table 3. A significant effect of the UFP is found. However, for control samples, the FE was constant. Increasing the SS proportion in the UF significantly decreased the FE. The most obvious reduction in the FE is found for the CTMP SS and then TMP SS. The FE of particleboards made with CTMP SS decreased by an average of 48.3 % compared to that of control panels. For the particleboards made with TMP SS, the FE showed an average decrease of 25.2 % compared to that of control panels. For those made with Kraft SS, the average decrease in FE was 6.7 % compared to that of control panels. These results indicate that the FE of particleboards made with SS as co-adhesive depends on its source.

Formaldehyde emission (FE) values from panels are shown in Table 6. Emissions from the normal UF proportion control panels (0 % SS) is comparable to values reported previously (Migneault et al. 2011a, b; Yang et al. 2006). Except for few experimental conditions (5 % UF and 100 % Kraft SS, 7 % UF and 75 % Kraft SS), the use of SS as co-adhesive led to an important decrease in FE (Table 6).

In good agreement with a recent report (Migneault et al. 2011a, b), these results suggest that beyond its use as co-adhesive, SS has proven its potential as formaldehyde scavenger for UF-bonded particleboard. The mechanism of FE reduction is still not fully understood but it could be linked to the higher proteins content in SS. Migneault et al. (2011b) also suggested that reactive functions in proteins in SS, such as amine and amide, captured free HCHO emitted from panels. Since the nitrogen content of Kraft was the lowest (1.3 %, crude protein = 6.25 × nitrogen), its ability to trap formaldehyde (HCHO) was the weakest, and this is due to its very high ash content (Table 2). Further investigations are needed.

In Table 6, it must be stressed that FE decreases as SS is added to UF, but the amount of UF does not decrease. In other words the decrease in FE is not due to replacement of UF by SS. Strangely, the FE does not increase with increasing UF content, but decreases markedly with SS content, and that is so, for all three sludges, especially TMP and CTMP. While increasing alkalinity due to added CTMP clearly interferes with mechanical and physical properties, it does not interfere with reduction in FE.

**Table 6** Formaldehyde emission (FE) ( $\mu\text{g/ml}$ ) from different particleboards (standard deviation in parentheses)**Tab. 6** Formaldehydabgabe (FE) ( $\mu\text{g/ml}$ ) verschiedener Spanplatten (Standardabweichung in Klammern)

UF (%)	SS/UF (%)	Pulping process					
		TMP		CTMP		Kraft	
		FE	Waller–Duncun test	FE	Waller–Duncun test	FE	Waller–Duncun test
5	0	0.25 (0.00)	a	0.25 (0.00)	a	0.25 (0.00)	abc
	75	0.19 (0.02)	cde	0.14 (0.01)	bcd	0.24 (0.02)	abc
	100	0.19 (0.01)	bcd	0.15 (0.00)	b	0.28 (0.02)	a
	125	0.17 (0.02)	de	0.14 (0.02)	bc	0.24 (0.01)	bc
7	0	0.24 (0.03)	ab	0.24 (0.03)	a	0.24 (0.03)	abc
	75	0.23 (0.06)	abc	0.13 (0.00)	bcd	0.25 (0.04)	ab
	100	0.18 (0.05)	de	0.12 (0.01)	bcd	0.24 (0.01)	abc
	125	0.18 (0.03)	de	0.11 (0.01)	d	0.22 (0.02)	bc
9	0	0.25 (0.01)	a	0.25 (0.01)	a	0.25 (0.01)	ab
	75	0.19 (0.01)	cde	0.14 (0.02)	bcd	0.21 (0.04)	c
	100	0.17 (0.02)	de	0.12 (0.02)	cd	0.23 (0.03)	bc
	125	0.14 (0.01)	e	0.12 (0.01)	cd	0.16 (0.02)	d

#### 4 Conclusion

Results from this study indicate that the use of SS from three different pulping processes as a co-adhesive has several advantages including the possibility of manufacturing particleboards at reduced UF resin content and wood particles content, value-added utilization of pulp and paper sludge and, especially, reduction in FE when the source and the percentage of SS were correctly chosen. The reduction in FE with the recycling of SS as co-adhesive is the most significant environmental benefit. Further investigations are needed to improve the thickness swelling of particleboards with SS as co-adhesive and to better understand the reaction between wood and UF with SS.

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