

CHARACTERISTICS OF PAPER MILL SLUDGE AND ITS UTILIZATION FOR THE MANUFACTURE OF MEDIUM DENSITY FIBERBOARD

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ABSTRACT

De-inking paper sludge (DPS) and primary sludge (PS) containing 20% secondary sludge from a paper mill were characterized as to their suitability for the manufacture of medium density fiberboard. Compared with DPS, PS had a lower ash content, higher holocellulose content, more and longer fibers, lower pH, and higher buffering capacity. These characteristics make PS a better fiber resource for fiberboard than DPS. Fiberboards were manufactured at the Pilot Plant of Forintek (Québec City, QC, Canada) using virgin spruce-pine-fir fiber (SPF) and PS or DPS at different sludge/SPF weight ratios with 12% urea-formaldehyde resin. At an equal sludge/SPF weight ratio, PS-SPF panels had much higher mechanical properties than did DPS-SPF panels. At a PS/SPF weight ratio of 7:3, the mechanical properties of PS-SPF panels were higher than the requirements of ANSI A208.2-2002 MDF standard for Grade 120 in terms of internal bond strength, modulus of rupture, modulus of elasticity, and thickness swelling. With DPS/SPF weight ratios as low as 3:7, the tested mechanical properties of DPS-SPF panels could meet the requirements of ANSI A208.2-2002 MDF standard for Grade 120.

Keywords: De-inking sludge, fiberboard, fiber properties, paper mill sludge, primary sludge, secondary sludge, spruce-pine-fir.

INTRODUCTION

The forest products industry relies heavily on the natural forest, but the pressure to reduce forest harvesting is forcing the industry to look for alternative fiber resources. Sludge is the final solid waste recovered from the wastewater treatment process in pulp and paper mills. The pulp

and paper industry in Canada produces about 7.1 dry Mt/y of sludge and most of the sludge is disposed through combustion and landfilling (Beauchamp et al. 2002). How to dispose of or make use of sludge has been a challenge for the pulp and paper industry. Some efforts have been made to use paper mill sludge for value-added products, such as gypsum fiberboards or fiberboard additives (Oztürk et al. 1992; Scott et al. 2000; Takats and Simatupang 1993; Davis et al. 2003). Environmental concerns and governmental regulations coupled with increasing demand

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for fiber materials are encouraging the forest products industry to utilize this potential fiber resource for wood fiber-based composite panels. Fiberboard panels are one of the most commonly used furniture and building materials, and the consumer demand for fiberboard panels has been increasing over the years.

Sludge discharged from paper mills is divided into four categories: 1) primary sludge (PS), which comes from the production of virgin wood fiber; 2) de-inking paper sludge (DPS), which comes from the process of removing inks from recycled paper; 3) secondary sludge (SS; activated sludge), which comes from the secondary wastewater treatment system; and 4) combined primary and secondary sludge. Total solid sludge produced by the Canadian pulp and paper industry comprises 42% PS, 12% DPS, 26% SS, and 18% combined sludge (Reid 1997). Primary sludge accounts for the majority of the total solid, and attempts have been made to use it for hardboard manufacture (Oztürk et al. 1992; Eroglu and Saatci 1993). Satisfactory hardboard was produced at a 1:4 weight ratio of PS to wood fiber. De-inking sludge has been characterized and investigated for use in building materials or fiberboard (Nadelman and Newton 1960; Davis et al. 2003). Secondary sludge has been excluded by many researchers because of its biological activity (Scott et al. 2000). However, it has been reported that hydrothermally-heated activated sludge could be used as a binder in the production of hardwood fiberboard (Lavrent'ev et al. 1978). The use of the treated activated sludge enhanced the flexural strength of the fiberboards by 23–32%.

Limited information is available on the suitability of sludge for the manufacture of fiberboard. In addition, the properties of fiberboard made from different kinds of sludge have not been compared. This study was intended for examining the suitability of various sludge for the manufacture of fiberboard. DPS and PS were obtained from a paper mill in Quebec City, Canada. The composition and fiber properties were analyzed and their suitability for the manufacture of fiberboard was evaluated in this study. The raw materials were refined, and fiberboard

panels were manufactured with or without virgin fibers at the MDF Pilot Plant of Forintek Canada Corp.

MATERIALS AND METHODS

Materials

De-inking paper sludge and PS were collected from Papiers Stadacona Ltée. in Quebec City, Canada. The PS was mixed with about 20% secondary sludge when it was de-watered in the paper mill. A urea-formaldehyde resin (UF-105) was obtained from Borden Chemical Canada Inc. (Quebec, Canada).

Preparation of raw materials for chemical analysis

The moisture contents of PS and DPS were determined according to ASTM standard (ASTM 1999). The sludge samples were then air-dried for one week and screened to remove plastic, glass, and other contaminating materials. The screened raw materials were ground in a Wiley mill fitted with a 35-mesh (0.5-mm) screen, and the portion that passed the screen was collected. The collected material was placed in an airtight container to balance the moisture content and then used for chemical analysis.

Determination of chemical composition of sludge

Ash content, klason lignin content, and holo-cellulose content were determined according to Tappi Standards (Tappi Test Methods 1993, Tappi Test Methods 1998, Tappi Useful Methods 249 1976).

Determination of pH and buffering capacity

The value of pH and buffering capacity were determined with a procedure similar to the previously reported method (Johns and Niazi 1980). In brief, the sludge sample (25 g, oven-dried) was refluxed in 250 ml of distilled water for 20 minutes and then filtered with Whatman #1 filter paper. The residue was washed with about 100

ml of distilled water, and the filtrate was made up to 500 ml with distilled water. Before the determination of pH and buffering capacity, the pH meter was calibrated with standardized buffer solutions at pH 4.0 and pH 7.0. For the determination of pH, 50 ml of the extract solution was pipetted into a 150-ml beaker. The pH value was recorded when the pH meter indicated a constant value. After pH determination, the extract solution was titrated with 0.025N NaOH solution to pH 8.0 for the determination of acid buffering capacity or with 0.025N H₂SO₄ solution to pH 3.0 for calculation of base buffering capacity. The acid buffering capacity is defined as the quantity (mmol) of NaOH required to raise the pH of the solution extracted from 100 grams of oven-dried sample to pH 8. Similarly, the base buffering capacity is defined as the quantity (mmol) of H₂SO₄ required to lower the pH of the solution extracted from 100 grams of oven-dried sample to pH 3.0.

Determination of fiber and particle size distribution

Primary sludge and DPS as received were suspended in water and then scanned with QualScanTM (McCarthy Products Company, Seattle, WA, USA) for fiber length and area distributions.

Refining of raw materials

For the dry process of manufacturing fiberboard, the moisture contents of the sludge samples as received were too high, and the sludge had to be dried before panel-making. Previous research showed that many clumps were formed after air-or oven-drying and the resulting clumps were not easy to break by blending. Therefore, DPS, PS, and spruce-pine-fir (SPF) samples were refined at steam pressure of 0.75 MPa with pre-heating retention time of 3 minutes and 2500 rpm of refiner speed at the MDF pilot plant of Forintek Canada Corp. in Quebec City, Canada. The refining process not only separated fibers and fines (short fibers and inorganic materials) very well, but also refined the knots in the sludge samples.

Manufacture of fiberboard panels

To investigate the effect of the weight ratio of SPF to sludge on panel properties, panels were manufactured using 100% SPF, 100% sludge, and SPF:sludge weight ratios of 3:7 and 7:3. The refined raw materials were mixed at the various DPS/SPF or PS/SPF ratios, and then 12% urea-formaldehyde resin (UF105) was applied to the dry fibers. For each condition, two panels of 610 mm × 610 mm × 10 mm and a density of 950 kg/m³ were made. Press temperature was set at 180°C and pressing time was 5 minutes.

Test of fiberboard panel properties

The panels were conditioned at 65 ± 1% relative humidity and 20 ± 3°C for one week. The internal bonding (IB), modulus of rupture (MOR), modulus of elasticity (MOE), and thickness swelling (TS) were tested according to ASTM standard (ASTM 1999).

RESULTS AND DISCUSSION

Characteristics of PS and DPS

The moisture contents of DPS and PS were 61.9% and 193.2%, respectively. As shown in Table 1, PS had a much higher moisture content than did DPS after having been air-dried for the same duration of time (one week). Primary sludge

TABLE 1. *Characteristics of primary sludge and de-inking paper sludge.*

	PS	DPS
Moisture, %	8.18	2.91
Ash content of oven-dried sample, %	19.50	53.62
Klason lignin content of oven-dried sample, %	27.84	25.69
Holocellulose content of oven-dried sample, %	62.73	42.88
pH	5.65	7.01
Acid buffering capacity, mmol NaOH/100 g o.d. sample	2.71	0.17
Base buffering capacity, mmol H ₂ SO ₄ /100 g o.d. sample	5.18	5.11

contained more klason lignin and holocellulose than DPS. However, DPS had a much higher ash content than did PS. Paper mill sludge is a mixture of mainly cellulose fibers and inorganic materials. A higher holocellulose content and a much lower ash content in PS indicate that it contained more cellulose fibers than did DPS. Cellulosic material absorbs more water than inorganic material, a factor that will directly affect the dimensional stability of fiberboards. PS had a lower pH and much higher acid buffering capacity than did DPS. Their base buffering capacities were comparable. A lower pH and higher acid buffering capacity of PS might provide a better environment for the curing of UF resin during the panel-making process than would DPS (Johns and Niazi 1980), which might further affect panel strength.

As illustrated in Fig. 1, DPS contained more fibers and particles with areas of $\leq 0.16 \text{ mm}^2$, but fewer fibers and particles with areas $\geq 0.29 \text{ mm}^2$ than did PS. More importantly, PS contained longer fibers ($\geq 0.54 \text{ mm}$) than did DPS (Fig. 2).

Effect of sludge and SPF weight ratio on internal bonding

As shown in Fig. 3, an increase in the weight ratio of PS to SPF resulted in a decrease in IB. Similarly, an increase in the weight ratio of DPS to SPF also resulted in a decreased IB. At the

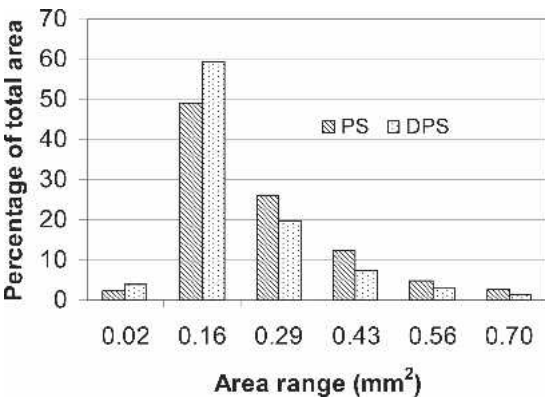


FIG. 1. Area distributions of fibers and particles in primary sludge and de-inking paper sludge.

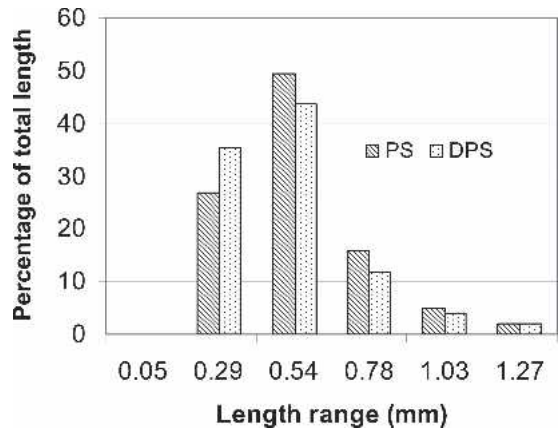


FIG. 2. Length distributions of fibers and particles in primary sludge and de-inking paper sludge.

same sludge/SPF ratio, PS-SPF panels had a higher IB than did DPS-SPF panels. To determine the relationship between IB and sludge proportion, IB was regressed against sludge percentage. The relationship of IB-sludge percentage was found to be linear and inverse with an R^2 as shown in Table 2. Internal bonding of the PS-only panels was higher than the requirement of ANSI A208.2-2002 MDF standard for Grade 130 (ANSI 2002).

A lower pH and higher acid buffering capacity of PS promote the development of IB by providing a better environment for the curing of UF resin during the pressing process. A higher

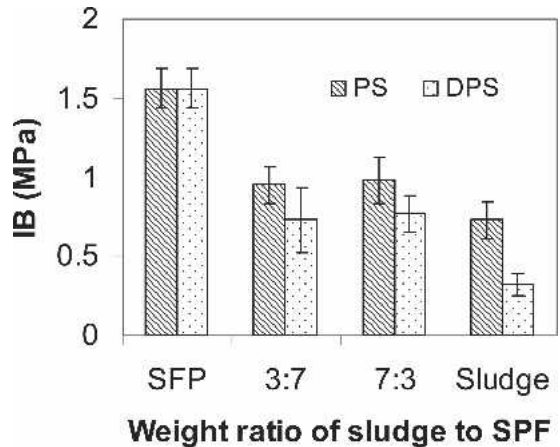


FIG. 3. Effect of the weight ratio of sludge to SPF on internal bonding.

TABLE 2. R^2 of linear regression of panel properties against sludge percentage of all fibrous raw materials (sludge and SFP).

	PS	DPS
Internal bonding	0.76	0.80
MOR	0.99	1.00
MOE	0.68	1.00
Thickness swelling	0.45	0.98

content of inorganic substances in DPS might obstruct the development of board strength (Grigoriou 2003). About 15–20% secondary sludge is always mixed with PS when it is de-watered and discharged by the paper mill. As stated above, secondary sludge contained in the received PS might contribute to the mechanical strength of PS-SPF panels.

Effect of sludge and SPF weight ratio on the panel MOR

The effect of the sludge and SPF weight ratio on the panel MOR is illustrated in Fig. 4. Increasing PS or DPS proportion in the panels resulted in a decreased MOR, and the decreases seen with DPS-SPF panels were more significant than those seen with PS-SPF panels. MOR was regressed against the sludge percentage of the fibrous raw materials, and the relationship of MOR-sludge percentage was found to be linear and inverse with a very high R^2 as shown in Table 2. At the same ratio of sludge to SPF,

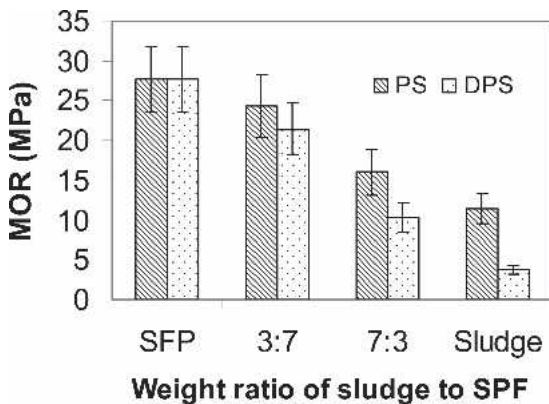


FIG. 4. Effect of the weight ratio of sludge to SPF on MOR.

PS-SPF panels had a higher MOR than did DPS-SPF panels. At a 7:3 of PS:SPF weight ratio, the MOR of PS-SPF panels was higher than the requirement of ANSI A208.2-2002 MDF standard for Grade 120.

Effect of sludge and SPF weight ratio on the panel MOE

As shown in Fig. 5, the MOE of PS-SPF and DPS-SPF panels decreased with increased weight ratio of sludge to SPF. MOE was regressed against the sludge percentage of fibrous raw materials. The relationship of MOE-DPS percentage was found to be linear and inverse with $R^2 = 1.00$, but the R^2 of the linear regression of MOE-PS percentage was only 0.68 (Table 2). At the same weight ratio of sludge to SPF, PS-SPF panels had a higher MOE than did DPS-SPF panels. Even if the panels were made with 100% PS as fibrous raw material, the MOE of the panels was still higher than the requirement of Grade 120 in ANSI A208.2-2002 MDF standard. To obtain a satisfactory MOE according to the same standard, as much as 30% DPS could be used as fibrous raw material.

Studies on fiber size distribution of PS and DPS indicated that PS contained more and longer fibers than did DPS. Cellulose fibers have a relatively high length-to-coarseness ratio, and much of the fiberboard strength is derived from the fibers. This explains why PS panels had

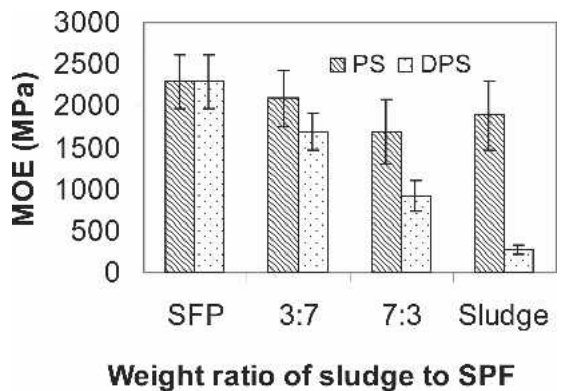


FIG. 5. Effect of the weight ratio of sludge to SPF on MOE.

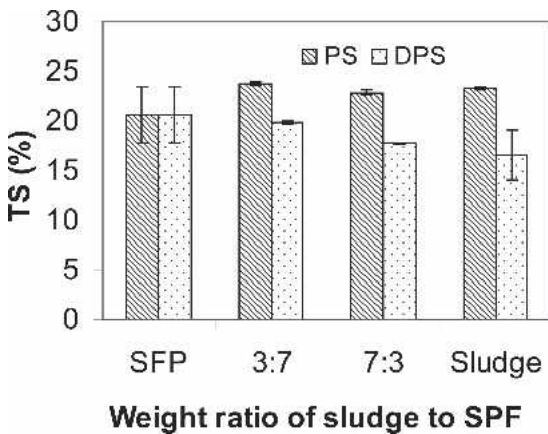


FIG. 6. Effect of the weight ratio of sludge to SPF on thickness swelling.

much higher MOR and MOE than did DPS panels when other process parameters were held constant.

Effect of sludge and SPF weight ratio on thickness swelling

As illustrated in Fig. 6, TS of DPS-SPF panels were slightly improved when DPS content increased. With the addition of PS to SPF, TS also increased, but the difference was not significant. Increase in PS content from 30% to 100% did not result in a further increase in TS. To determine the relationship between TS and the weight ratio of sludge to SPF, TS was regressed against the sludge percentage. The relation between TS and DPS percentage was linear with $R^2 = 0.98$, but the R^2 of linear regression on TS against PS percentage was only 0.45 (Table 2). PS-SPF panels had a higher TS than did DPS-SPF panels at the same weight ratio of sludge to SPF. This could be explained by the difference in their chemical composition. A higher mineral content and a lower holocellulose content in DPS has been shown to contribute to the reduced swelling of manufactured boards (Grigoriou 2003).

In comparison, TS of all panels were slightly higher than the requirement of ANSI A208.2-2002 MDF standard. However, this problem could be solved by the application of wax.

CONCLUSIONS

Primary sludge mixed with 20% secondary sludge contained more and longer fibers than did DPS, and it had a much lower ash content. Compared to DPS, PS also had a lower pH but higher acid buffering capacity. These properties made PS more suitable as a fiber material for the manufacture of fiberboard than DPS. At the equal weight ratio of sludge to SPF, MDF panels made of PS and SPF had much higher mechanical properties than did DSP-SPF panels. The dimensional stability could be further improved by the application of wax. With 12% UF resin, it is possible to replace 70% of SPF fiber with PS fiber or 30% of SPF fiber with DPS fiber in the manufacture of MDF and the panel properties meet the ANSI A208.2-2002 standard for Grade 120. This study showed that PS had an excellent potential for the manufacture of fiberboard.

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