HEXAMETHYLDISILAZANE TREATMENT TO RESTORE STRENGTH OF RECYCLED FIBER

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ABSTRACT

When pulp fibers dry, many fibrils and microfibrils dry down onto the surface of the fiber and are unavailable for fiber bonding. Therefore, the paper or paper products produced are not as strong as similar products made from pulp that has never been dried. Recent studies have suggested that hexamethyldisilazane (HMDS) treatment of pulp fibers will raise dried-down fibrils and microfibrils. The objective of this work was to determine if HMDS treatment can be used to increase sheet strength of fiber from recycled corrugated fiberboard components by raising fibrils and microfibrils from fiber surfaces. Overall, HMDS treatment improved burst, tear, and tensile indices of handsheets from recycled fibers from linerboard and corrugated medium. Scanning electron micrographs show the presence of many more raised fibrils and microfibrils on HMDS-treated fiber. These raised fibrils and microfibrills appear to be the cause for increased sheet strength.

Keywords: Hexamethyldisilazane treatment, recycled fibers, fibrils, microfibrils, scanning electron microscopy, strength properties, bonding.

INTRODUCTION

When pulp fibers dry, fibrils and microfibrils dry down onto the fiber surface, and these dried-down fibrils and microfibrils remain unavailable for interfiber bonding even upon rewetting (Clark 1978). Recycled fiber, such as from old corrugated containers, does not produce paper or paperboard products that are as strong as similar products made from pulps that have never been dried (Horn 1975; Klungness 1974; Koning and Godshall 1975; Maltenfort 1970; McKee 1971). Thus recycled fiber is generally limited to lower strength products. In order to increase the use of recycled fiber, we need to establish ways to increase strength by enhancing interfiber bonding.

Recent studies have suggested that hexamethyldisilazane (HMDS)-treated pulp fibers: (1) retain many fibrils and microfibrils in a raised position on the fiber surface during drying, and (2) will raise fibrils and microfibrils that have been dried down on fiber surfaces (Sachs 1986; 1987). Therefore, the objectives of this work were to: (1) demonstrate that HMDS treatment can be used to increase strength of recycled corrugated fiberboard, and (2) show the presence of raised fibrils and microfibrils on fiber surfaces by use of scanning electron microscopy (SEM). Because corrugated fiberboard consists of kraft linerboard and semichemical corrugated medium, these two components were investigated separately.

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MATERIALS AND METHODS

Fiber furnish

Corrugated fiberboard sheets were obtained from a single lot of commercial singlewall corrugated fiberboard. The fiberboard consisted of two 205-g/m^2 linerboards and one 127-g/m^2 corrugated medium. The corrugated fiberboard was soaked in 23.8 C water for 3 h to separate the linerboard from the corrugated medium. The medium was then placed in a British Disintegrator for 750 revolutions.[2] The linerboard was pulped in the British Disintegrator for 1,000 revolutions. Each was separately dewatered on a Buchner funnel. Samples of the dewatered linerboard and medium were independently refined in a PFI mill to nominal Canadian Standard Freenesses (CSF) of 500, 375, and 280 ml for the corrugated medium and 640, 495, and 360 ml for the linerboard. Samples were tested with iodine and found to be free of starch.

HMDS treatment

Fibers from the refined linerboard and corrugated medium sample furnishes were treated with HMDS. HMDS-treated fibers were prepared by dehydrating the wet fiber through a series of ethanol solutions of 70%, 85%, 95%, and 100%, with 5 minutes stirring in each solution.[3] After the absolute ethanol treatment, the fibers were immersed in HMDS. The HMDS was then allowed to evaporate overnight from the fibers in a hood at room temperature. A comparison series not treated with HMDS was also prepared.

Handsheet preparation and strength measurements

Handsheets from the different freeness HMDS-treated and nontreated fibers were prepared according to TAPPI Standard T205. Tests were made on 60-g/m^2 handsheets made from the above described fiber samples. Burst, tensile, and tear indices on handsheets were measured according o TAPPI procedures T403, T404, and T470, respectively.

Microscopy

Fiber specimens were mounted on aluminum stubs and gold coated to approximately 100 Å thickness for SEM. The gold-coated fibers were studied and photographed using a JEOL 840 scanning electron microscope at an accelerating voltage of 15 kV. Non-HMDS-treated pulp fibers were dried by critical point drying,[4] which consists of dehydrating pulp fibers with a solvent (i.e., ethanol) followed by replacing the dehydrating solvent in the cell walls of the pulp fibers with a transitional fluid (i.e., liquid carbon dioxide) (Cohen 1974). Subsequently, the transitional fluid is brought to its critical point: a temperature and pressure at which the densities of the liquid and the gas of the fluid are identical. As a

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[2] The use of trade or firm names in this publication is for reader information and does not imply endorsement by the U.S. Department of Agriculture of any product or service.

[3] Drying fibers from ethanol by itself in the absence of HMDS does not raise fibrils and microfibrils.

[4] Because wet fibers cannot be used in the vacuum environment of the SEM, the wet (no HMDS treatment) fibers were critical point dried. Critical point drying has been found to be an acceptable method of preserving wet fiber morphology and is a commonly used technique in electron microscopy.

TABLE 1. *Properties of 60 g/m² handsheets made from HMDS-treated and nontreated linerboard and corrugated medium.*[a]

	CSF (ml)	Burst index (kPa·m²/g)	Tensile index (Nm/g)	Tear index (mN·m²/g)	Density (kg/m³)
		Linerboard			
Nontreated	640	2.67	49.2	12.83	521
	495	4.29	68.9	11.06	582
	360	4.70	79.6	9.85	611
HMDS-treated	670	3.48	57.5	13.01	506
	590	4.79	75.6	11.70	562
	450	5.31	80.0	10.88	591
		Corrugated medium			
Nontreated	500	1.88	41.1	7.07	522
	375	2.43	55.8	6.98	559
	280	3.06	60.8	6.52	578
HMDS-treated	500	2.06	53.2	7.40	532
	380	3.12	61.2	7.54	563
	315	3.43	65.7	7.60	582

[a] HMDS = Hexamethyldisilazane.

result, the liquid in the fiber cell wall passes from the liquid phase to the gaseous phase without exerting surface tension on the pulp fibers (Lewis and Nemonic 1973).

RESULTS AND DISCUSSION

Handsheet strength properties

At a given initial freeness, burst, tensile, and tear indices of 60-g/m² handsheets produced from HMDS-treated recycled linerboard fiber and corrugated medium fiber were greater than those from their respective handsheets prepared from nontreated fiber (Table 1). As freeness decreased, burst and tensile indices in-

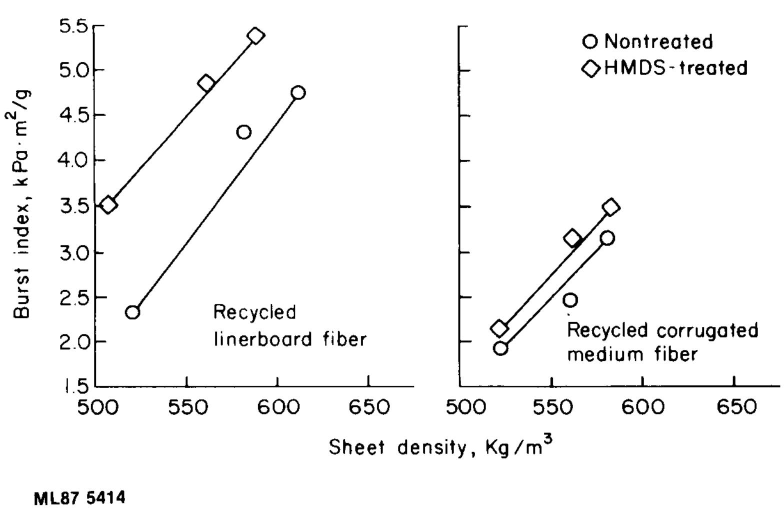

FIG. 1. Burst index versus sheet density for recycled fiber. HMDS = hexamethyldisilazane. (ML87 5414)

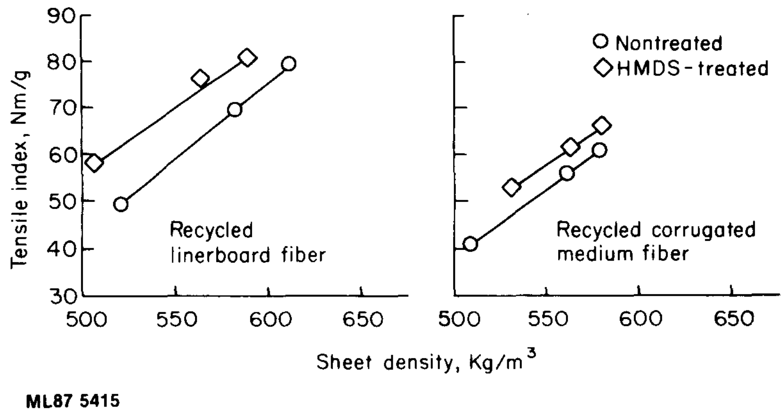

FIG. 2. Tensile index versus sheet density for recycled fiber. HMDS = hexamethyldisilazane. (ML87 5415)

creased, while tear index generally decreased. In addition, HMDS treatment generally increased pulp freeness versus the nontreated fiber. The increase in freeness was greater with the recycled linerboard than with the recycled corrugated medium and was largest at the lowest freeness.

Handsheet strength properties plotted versus sheet densities are shown in Figs. 1 and 2. At a given sheet density, the HMDS-treated fiber gave higher burst and tensile indices than the respective nontreated fiber. The absolute increase in burst and tensile index values brought about by HMDS treatment was larger for the recycled linerboard fiber, which is produced from softwood kraft pulp, than for the corrugated medium, which is produced from semichemical hardwood pulp. For the recycled linerboard, HMDS treatment increased burst index by approximately 1.25 kPa m^2/g (up to 1.7×) and tensile index by approximately 10 Nm/g (up to 1.2×). HMDS treatment of recycled corrugated medium increased burst index by approximately 0.25 kPa m^2/g (up to 1.2×) and tensile index by approximately 5 Nm/g (up to 1.1×).

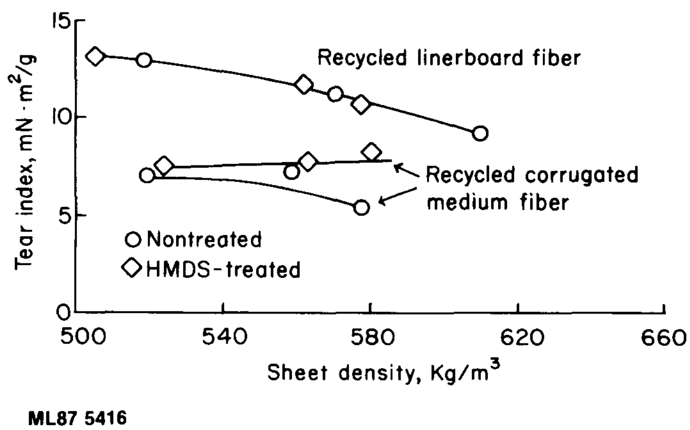

FIG. 3. Tear index versus sheet density for recycled fiber. HMDS = hexamethyldisilazane. (ML87 5416)

At a given sheet density, HMDS treatment increased the tear index values (up 1.7×) of sheets produced from recycled corrugated medium (Fig. 3). HMDS treatment did not, however, increase the tear index of sheets produced from recycled linerboard. This difference may be due to differences in fiber morphology and pulp fiber yield. Length of softwood pulp fibers used in linerboard is about three times that of hardwood pulp fibers used in corrugated medium (Rydholm 1965). In addition, pulp fiber used in corrugated medium is produced by high-yield semichemical pulping while pulp fiber used in linerboard is produced by low-yield kraft pulping. Therefore, improved interfiber bonding brought about by HMDS treatment may be more effective with recycled corrugated medium in improving tear index.

The overall increase in sheet strength with HMDS treatment may be explained as being caused by increased interfiber bonding brought about as a consequence of raising fibrils and microfibrils from fiber surfaces. SEM was used to examine fiber surfaces of recycled linerboard and corrugated medium before and after HMDS treatment.

Examination of fiber surfaces

HMDS treatment of fibers from recycled linerboard and corrugated medium was effective in raising fibrils and microfibrils from fiber surfaces. SEM micrographs of HMDS-treated and nontreated linerboard fibers (Figs. 4a, b) show the presence of raised fibrils and microfibrils. However, there are many more fibrils and microfibrils in a raised position on the HMDS-treated fibers (Fig. 4a). SEM micrographs of the HMDS-treated and nontreated corrugated medium were similar (Figs. 5a, b). At each freeness level, the HMDS-treated recycled linerboard and corrugated medium fiber showed the presence of more raised fibrils. Hence, increases in burst and tensile indices may be explained by the increased number of raised fibrils and microfibrils available for interfiber contact. Therefore, HMDS treatment of recycled fibers from linerboard and corrugated medium appears to be a satisfactory method for raising fibrils and microfibrils and improving overall strength.

The manner in which HMDS works on fibers other than raising fibrils and microfibrils has not been investigated. HMDS is a reagent commonly used in gas chromatography primarily to prepare silyl ethers of compounds with one or more reactive hydrogen atoms such as sugars, amino acids, alcohols, and other compounds (Nation 1983). It is not known whether HMDS reacts with similar chemical groups in the fibers themselves and thus affects other factors such as interfiber bond strength and fiber flexibility. This aspect as well as other mechanisms involved will be investigated in future work.

CONCLUSIONS

HMDS treatment of recycled fiber from linerboard and corrugated medium offers a way to substantially increase sheet strength. At a given sheet density, increases in burst and tensile index values with HMDS treatment were greater for the recycled fiber from linerboard than for the corrugated medium. With tear index, however, HMDS treatment increased tear index values of sheets produced from recycled corrugated medium while it did not with recycled linerboard fiber. SEM showed the presence of many more raised fibrils and microfibrils from fiber

FIG. 4. SEM micrographs of recycled fiber from linerboard. Top: Hexamethyldisilazane-treated, 590 CSF, 700×; Bottom: nontreated, 495 CSF, 700×. (MC87 9010)

FIG. 5. SEM micrograph of recycled fiber from corrugated medium. Top: Hexamethyldisiazane-treated, 380 CSF, 700×; Bottom: nontreated, 375 CSF, 700×. (MC87 9009)

surfaces following HMDS treatment. Raised fibrils and microfibrils from fiber surfaces appear to increase interfiber bonding and thus bring about the increases in strength following HMDS treatment of fiber.

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