RETAINING RAISED FIBRILS AND MICROFIBRILS ON OAK FIBER SURFACES

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ABSTRACT

Drying of spruce softwood kraft fibers treated with hexamethyldisilazane (HMDS) has been found to: (1) maintain fibrils and microfibrils in a raised position on fiber surfaces, (2) raise fibrils and microfibrils that had been dried down on fiber surfaces, and (3) increase strength properties of handsheets made from dried fiber. The question whether shorter thick-walled hardwood fibers behave similarly is examined in the present study of the effect of HMDS-drying on oak kraft pulp fibers. The effects of HMDS-drying, air-drying, and paper-machine-drying were evaluated by observing the surfaces of refined hardwood kraft pulp fibers. Using scanning electron microscopy, the fiber surfaces of these dried fibers were compared with those of never-dried fiber whose morphology was preserved by critical point drying. In addition, air-dried and paper-machine-dried fibers were examined after rewetting and HMDS-treatment to recover fibrils and microfibrils. Overall strength properties were considerably greater when handsheets were made from pulps treated with HMDS than from pulps that were dried in air or on the paper machine.

Keywords: Hexamethyldisilazane treatment, air-drying, paper-machine-drying, bonding, fibers, fibrils, microfibrils, hardwood pulp, scanning electron microscopy, strength properties.

INTRODUCTION

Pulp produced from hardwoods is different from pulp produced from softwoods (Thomas 1970). Softwoods have a uniform arrangement of a few cell types. Hardwoods, in contrast, are composed of widely varying proportions of different kinds of cells. Tracheids, vessel elements, longitudinal parenchyma, and ray parenchyma make up a significant volume in most hardwoods (McIntosh 1970). Longitudinal cells of hardwood vary and are considerably shorter than the longitudinal cells of softwoods.

Usually, the shorter, thick-walled fibers of hardwood such as oak make paper with lower burst, tear, and tensile strengths than softwood fibers (Britt 1970). Paper strength properties depend on a high degree of fiber-to-fiber bonding (Britt 1970). Moreover, the presence on fiber surfaces of raised fibrils and microfibrils available for interfiber bonding is necessary for strength of the paper products (Robinson 1980). Therefore, it is important to keep fibrils and microfibrils from drying down on the fiber surfaces.

In previous work, I reported that fibrils and microfibrils on softwood (spruce kraft) fiber surfaces could be satisfactorily maintained in the raised position fol-

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lowing critical point drying (Sachs 1985). Albrecht and MacKenzie (1975) reported that it is possible satisfactorily to maintain biological cells in a near-natural state by drying them with organic solvents (ethanol or acetone). However, long periods of drying (24–48 hours) at low temperatures (−30 C to −40 C) were required for optimal results. I have also demonstrated that treatment with hexamethyldisilazane (HMDS) solvent offers a way to dry softwood (spruce kraft) pulp fibers without causing fibrils and microfibrils to dry down upon fiber surfaces (Sachs 1986). Overall, handsheets had greater strength properties when made with pulp treated with HMDS than with pulps dried in air or by paper machine (pulps in which fibrils dried down on fiber surfaces). HMDS-drying of spruce kraft pulp fibers was a rapid procedure—usually 25 minutes at room temperature.

HMDS-drying of spruce softwood kraft fibers was found to: (1) maintain fibrils and microfibrils in a raised position on fiber surfaces, (2) raise fibrils and microfibrils that have been dried down on fiber surfaces, and (3) increase strength properties of handsheets made from dried fiber. The question whether shorter thick-walled hardwood fibers would behave similarly is examined in the present study of the effect of HMDS-drying on oak kraft pulp fibers. The effects using HMDS, air and paper machine for drying were evaluated by observing the surfaces of refined hardwood kraft pulp fibers. The fiber surfaces of these dried fibers were compared with those of never-dried fiber using scanning electron microscopy (SEM). In addition, air-dried and paper-machine-dried fibers were examined after rewetting and using HMDS-drying to recover fibrils and microfibrils. Handsheets (205 g/m^2) were prepared from pulps before (never-dried) and after treatment with each drying procedure. Strength properties measured were burst, tear, tensile, and compression indices.

EXPERIMENTAL

Fiber furnish

White oak (*Quercus alba* L.) unbleached kraft pulp (45% yield) was refined in a PFI mill to nominal Canadian Standard Freenesses (CSF) of 560 ml, 400 ml, and 265 ml.

Air-dried fibers were prepared by drying never-dried fiber at 20 C and 27% relative humidity. Thermal-dried fibers (paper-machine-dried) were prepared by forming never-dried fiber into 205-g/m^2 webs and drying them on a drum dryer at a temperature of 120 C. With HMDS-drying, never-dried fibers were first dehydrated, through a series of ethanol solutions of 70%, 85%, 95%, and 100%, with 5 minutes stirring in each. After the absolute ethanol treatment, the pulp fibers were immersed in HMDS for 5 minutes. The HMDS was then allowed to evaporate from the fiber in a hood at room temperature. (Note: Drying fibers from ethanol by itself in the absence of HMDS does not preserve raised fibrils and microfibrils.)

Handsheet preparation and strength measurements

Tests were made on 205-g/m^2 handsheets made from the above-described types of dried pulp samples as well as from never-dried pulp. Preparation of handsheets followed the procedure of Tappi T205. In evaluations where HMDS was used to recover raised fibrils and microfibrils from fiber surfaces, handsheets were made from paper-machine-dried webs and air-dried fibers that were shredded and re-

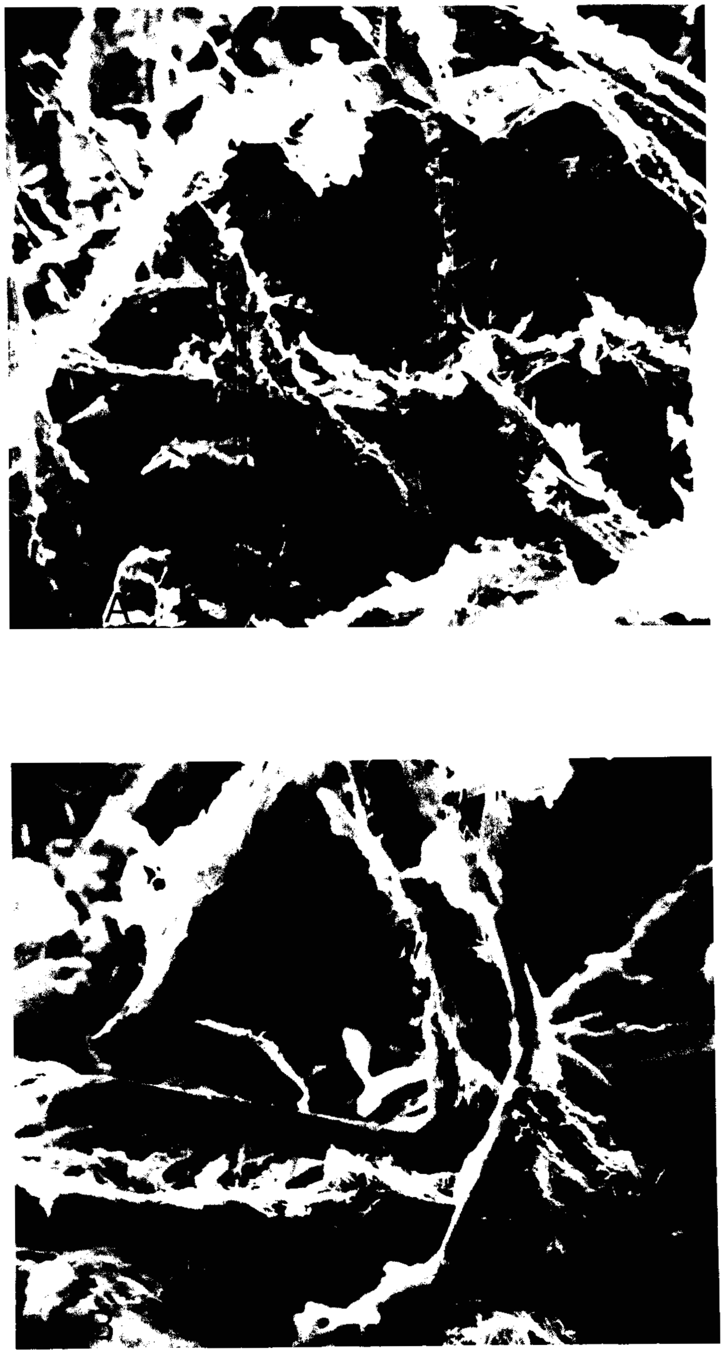

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Fig. 1. Red oak kraft pulp. (A) HMDS-treated. 200×. (B) Never-dried. 200×.

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FIG. 2. Red oak air-dried. 200×.

wetted by slushing in a British Disintegrator at 500 revolutions. The rewetted pulp fiber was then dewatered on a Buchner funnel and HMDS-dried as described above.

Strength tests on handsheets followed the procedures of Tappi: burst index—T403; tensile index—T404; tear index—T470. Compression tests were performed according to the method of Gunderson (1983).

Microscopy

Pulp fiber specimens, dried by the various drying techniques, were mounted on aluminum stubs and gold coated approximately 100 Å thickness for SEM observations. The gold-coated pulp fibers were studied and photographed using a Cambridge Mark II scanning electron microscope at an accelerating voltage of 20 kV. Never-dried fibers were also observed in the SEM. Because wet fibers cannot be used in the vacuum environment of an SEM, the never-dried fibers were at their critical point (Sachs 1985). Drying to the critical point has been found to be an acceptable method of preserving never-dried fiber morphology and is a commonly used technique in electron microscopy (McIntosh 1970).

RESULTS AND DISCUSSION

Preserving and recovering raised fibrils and microfibrils

Scanning electron micrographs of HMDS-dried oak kraft pulp (Fig. 1A) and never-dried oak kraft pulp (Fig. 1B) display raised fibrils and microfibrils on the

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FIG. 3. Red oak kraft pulp fibers in water. (A) Never-dried. Note presence of many fibrils (arrow). 310×. (B) Air-dried. Note lack of fibrils. 310×.

surfaces of fibers. In contrast, air-dried (Fig. 2) and paper-machine-dried oak kraft pulp fibers show few fibrils between adjacent fibers or in a raised position from the fiber surface. Air-dried and paper-machine-dried oak kraft pulp fibers, upon rewetting, recover few raised fibrils from fiber surfaces as compared to never-dried pulp fibers (Fig. 3). However, air-dried and paper-machine-dried oak kraft pulp fibers, rewetted and HMDS-dried, regain some of the dried-down fibrils and microfibrils from the fiber surface (Fig. 4) but not as many as found on never-dried pulp fibers (Fig. 1B).

The effects of HMDS treatment appear similar in spruce (Sachs 1986) and in oak kraft pulps not only in preserving raised fibrils and microfibrils upon drying but also in recovering raised fibrils and microfibrils from the surface of previously air-dried or paper-machine-dried pulp fibers.

Strength properties

At comparable freeness, strength properties (burst, tear, tensile, and compression indices) were nearly equal, whether handsheets were produced from HMDS-dried pulp fiber or never-dried pulp fiber (Tables 1 and 2). Overall, handsheets produced from oak kraft pulp fibers that were HMDS-dried had considerably greater strength properties than those from fibers that were air-dried or paper-machine-dried. When the fibers were paper-machine-dried, handsheets had lower strength properties than when they were dried in air. As pulp freeness decreased, handsheet strength properties, expressed as a percentage of handsheet strength property for the same freeness produced from never-dried pulp, generally increased with all drying methods.

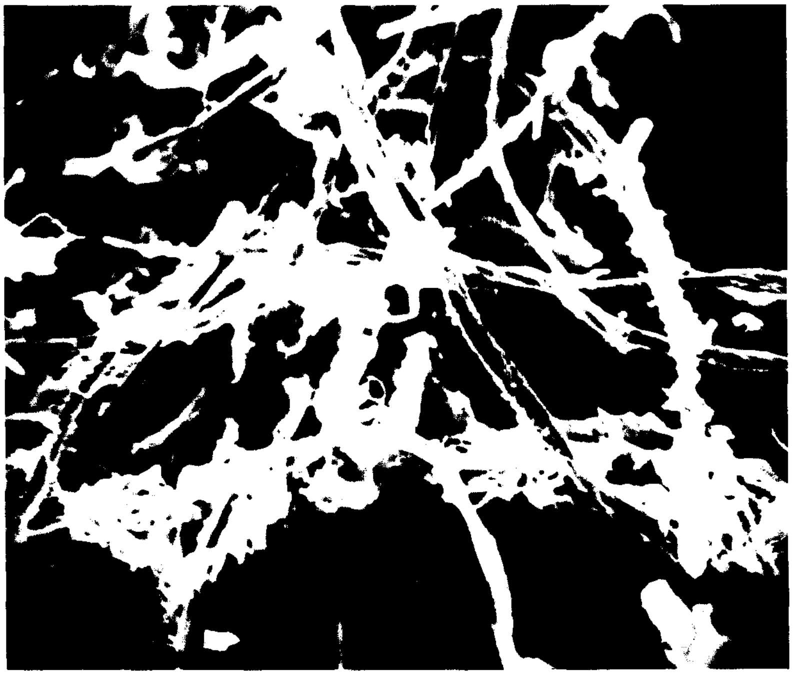

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FIG. 4. Red oak kraft pulp air-dried, rewetted, and HMDS-treated, 200×.

In maintaining never-dried properties, HMDS appears to have brought about a fundamental change in oak kraft pulp properties. The increase in handsheet strength properties may possibly be explained as a consequence of keeping the fibrils and microfibrils raised from the fiber surface so that interfiber bonding can take place.

Comparison of strength properties can be made between handsheets made from pulp fibers where fibrils and microfibrils have been dried down upon fiber surfaces and then raised using HMDS treatment and handsheets made from fibers where raised fibrils and microfibrils were preserved during drying. This is done by comparing handsheets made from air-dried or paper-machine-dried, rewetted, HMDS-dried fiber to handsheets made from HMDS-dried fiber (Table 2). All strength indices were generally much lower for the rewetted fiber. HMDS drying, although it raised some of the fibrils and microfibrils dried down on fiber surfaces,

TABLE 1. *Properties of 205-g/m² handsheets made from never-dried unbleached oak kraft pulps.*

Canadian standard freeness, ml	Burst index, kPa-m²/g	Tear index, mN-m²/g	Tensile index, Nm/g	Compression index, kN/m	Density, kg/m³
560	6.00	10.3	82.0	6.37	698
400	7.40	10.6	93.4	6.91	780
265	7.80	10.2	92.9	6.53	810

TABLE 2. *Effect of HMDS treatment, air drying, and paper machine drying on 205-g/m² handsheet properties.*[1]

Method of drying	Freeness, ml	Burst index, %	Tear index, %	Tensile index, %	Compression index, %
	Unbleached oak kraft				
HMDS	560	84.2	87.7	88.8	95.4
	400	90.1	100.0	90.4	93.2
	265	89.7	107.8	96.1	107.0
Air	560	52.0	72.5	62.0	74.5
	400	63.5	96.2	68.8	79.3
	265	67.3	100.9	72.0	89.2
Paper machine	560	35.8	61.2	49.5	59.8
	400	45.1	83.8	56.2	72.9
	265	54.6	87.4	61.1	78.5
Air-dried, rewetted HMDS	560	57.8	80.2	64.0	78.2
	400	60.9	93.8	66.5	82.2
	265	67.3	104.9	74.9	87.6
Paper-machine-dried, rewetted HMDS	560	42.5	73.6	55.6	71.1
	400	51.3	88.2	62.1	79.3
	265	62.4	104.9	63.3	83.9

[1] Expressed as percent of handsheet property produced from never-dried fibers at the same freeness (Table 1).

did not substantially improve the strength properties of air-dried or paper-machine-dried pulps.

A further comparison of the effectiveness of HMDS in maintaining strength properties can be made between handsheets prepared from HMDS-dried softwood (spruce) (Sachs 1986) and hardwood (oak) kraft pulp fiber. HMDS treatment appeared to be equally effective in maintaining strength properties whether the handsheets were prepared from hardwood or softwood kraft pulp fiber or from their respective never-dried pulp fibers. Additionally, softwood and hardwood kraft pulp fiber gave handsheet strength properties considerably greater when HMDS-dried than when air-dried or paper-machine-dried. When fibrils and microfibrils had been dried down on fiber surfaces (i.e., with air-dried and paper-machine-dried fiber), HMDS treatment was less effective in improving handsheet strength properties with hardwood than softwood kraft pulp fiber. This dissimilarity may be an expression of fiber morphology of the hardwoods that require considerably more beating than softwoods to enhance fiber bonding (Rydholm 1965).

CONCLUSIONS

HMDS-drying offers a way to dry oak kraft pulp fibers without causing many fibrils and microfibrils to dry down upon fiber surfaces. Overall, strength properties are considerably greater when handsheets are made from pulps treated with HMDS than from pulps dried in air or by paper machine. For dried fibers, on which fibrils and microfibrils have been dried down on the fiber surface (i.e., air-dried or paper-machine-dried), rewetting, followed by HMDS-drying, raises some of the fibrils and microfibrils from the fiber surface. The effects of treatment with HMDS are similar in softwood (spruce) kraft pulp and hardwood (oak) kraft pulp in preserving raised fibrils and microfibrils and maintaining handsheet strength properties. HMDS treatment of air-dried and paper-machine-dried hardwood (oak) kraft pulp is less effective in improving strength properties than when it is applied to softwood (spruce) kraft pulp fiber.

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