STRENGTH LOSS IN RECYCLED FIBERS AND METHODS OF RESTORATION

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ABSTRACT

The reduced interfiber bonding capability and reduced conformability of recycled fibers compared to virgin wood pulp fibers is caused by the drying phase of the first papermaking cycle. Changes in the fiber result in stiffness. This effect is more pronounced in chemical pulps than in high lignin content mechanical pulps. This chapter describes methods for restoring some or all the interfiber bonding. In an attempt to develop a "dry" newspaper recycling process, the water-intensive repulping and paper-forming steps were replaced with dry-fiberizing, air-forming, gas-phase ozone and ammonia treatments, and press-drying. The tensile strength of the dry-recycled paper approached that of the original newsprint.

INTRODUCTION

The fundamental changes in paper properties that occur during recycling are discussed in detail in Chapter 16. In this chapter, we discuss the factors that lead to a reduction in interfiber bonding. Methods of restoring some or all the original interfiber bonding are then categorized and briefly discussed. Within this framework, we describe the results of a research program to enhance the interfiber bonding of dry-fiberized newsprint pulp. The program was part of an attempt to develop a process for a small urban mill to recycle newspapers into newsprint with a minimum of water usage.

PROPERTY CHANGES

Interfiber Bonding Capacity

The most generally recognized property losses in paper recycling are those associated with interfiber bonding, such as tensile and burst strengths. Interfiber bonding consists primarily of hydrogen bonds between the surface carbohydrate macromolecules of neighboring fibers. The extent and magnitude of interfiber bonding depends on the exposure of polysaccharide molecules and the surface functional groups such as hydroxyl, carbonyl, and carboxyl. It also depends on the extent of surface contact between fibers.

The property that reflects the ease with which fibers are flattened and brought into contact with one another is known as fiber conformability. Surface contact may also be increased by fibrillation. Fibrillation is a disruption of the surface structure of the fiber, which produces strands or ribbons of cell wall polysaccharides (see Chapters 4 and 18). When the fibers are laid down in a paper mat, this fibrinous material overlaps adjacent fibers, creating strong interfiber bonds.

Loss in interfiber bonding is observed in fibers that have been thoroughly dried, as in the dryer section of most paper machines. The loss is attributed to collapse of polysaccharide macromolecules onto each other as a result of dehydration, resulting in strong intermolecular hydrogen bonds. This general process has been termed hornification. Hornified fibers

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are stiffer because of internal collapse and are more resistant to fibrillation because of the strong intrafiber bonding of polysaccharides at or near the fiber surface.

Chemical pulps, particularly bleached chemical pulps, are more susceptible to hornification than are mechanical pulps because the removal of lignin provides the opportunity for greater intermolecular mobility and contact within the carbohydrate component. Also, fibers with high lignin content are initially more rigid because of the three-dimensional structure and crosslinking characteristics of the lignin macromolecule, so initial strength is reduced. Under mild drying conditions, the flexibility of fibers with high lignin content has been shown to increase upon repeated cycling; the greater flexibility results in increased conformability and interfiber bonding (Chapter 16).

Internal Fiber Stiffening

Internal fiber stiffening is interrelated with the surface effects that affect interfiber bonding. When horrification occurs with molecules internal to the fiber, the resultant stiffer fiber is not as conformable to other fibers and there is less opportunity to bond. The internal hornification is quite similar to the phenomenon on the surface with respect to the tight aggregation of the cell wall polysaccharides, but it also includes the possibility of crystallization of the cellulose and the hemicelluloses within the fiber cell walls. These processes, which are discussed in greater detail in Chapter 18, are essentially the result of the annealing of the cellulose as a result of exposure to elevated temperatures during the drying process. Although this exposure is of relatively short duration during the papermaking cycle, it has been shown to be sufficient for some crystallization to occur because of the enhanced mobility of the cellulose when well saturated with water [1]. When wet annealing is followed by dehydration, the enhanced crystallinity imparts greater stiffness to the fibers, not unlike that which might result from a limited amount of crosslinking. The crystallization may in fact be viewed as a physical crosslinking process. As mentioned earlier, the primary impact of this physical transformation is stiffening of the fibers, which results in reduced conformability during the papermaking process.

Fiber Shortening

An increase in fines is usually observed during the recycling operation (Chapter 3). The fines may result from the cutting of fibers or from fibrillar debris from previously bonded paper. The amount of fines generated depends on the severity of the original papermaking process and the extent of mechanical treatment during recycling. Under controlled conditions with gentle drying and beating, the breakdown of fibers has not been detectable (Chapter 16). A possible reason for this result, in contrast to that usually observed in commercial practice (Chapter 3), is that the drying operation may contribute significantly to the generation of fines in recycling. The relation of fiber length to paper tensile (bonding) strength is somewhat complex. Fines may have a beneficial effect on interfiber bonding. However, if carefully fractionated fibers are cut so that fiber length is the only variable, a decrease in tensile strength is observed [2]. When paper is dry-fiberized, the shortening of fibers and generation of fines are very marked, as will be discussed later.

INTERFIBER BONDING ENHANCEMENT

At least six options are available to mills to enhance the strength properties of papers made from recycled fibers: mechanical treatment, blending with virgin fibers, chemical additives, fractionation, chemical treatment, and paper machine process modifications.

For recycled mechanically pulped fibers, a mechanical treatment such as disk refining restores nearly all the original strength. However, unless special efforts are made to reduce the refining intensity, the strength gain may come at a sacrifice of drainage because of the generation of fines. Doshi provided suggestions for treating fibers [3]. Softwood kraft

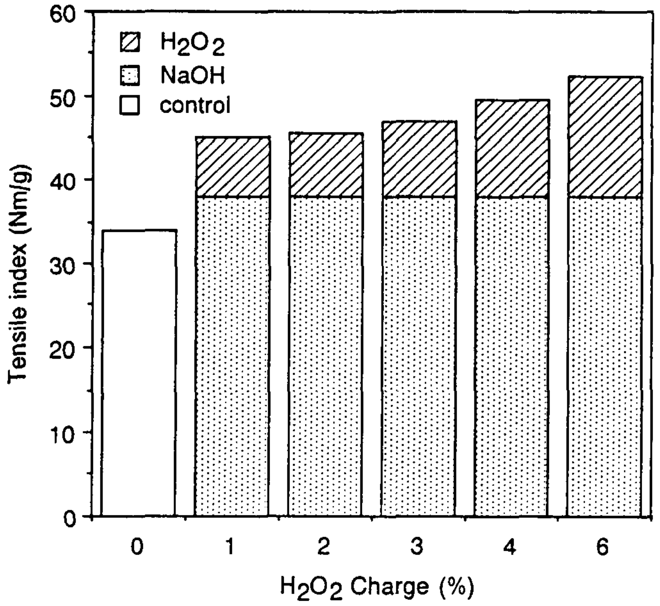

Figure 1—Effect of H_2O_2 and NaOH on tensile index of wet-fiberized newsprint (70 g/m^2 handsheets).

pulps may also be refined to the initial strength level. However, the more the initial pulp is refined, the more difficult the restoration of initial strength with mechanical treatment alone [4].

Although a few mills are producing 100% recycled paper, the majority are attempting to meet recycling demands by adding recycled fibers to their present virgin fiber lines. In this regard, the question is usually how much recycled fiber can be accommodated rather than how much virgin pulp is required to upgrade the recycled pulp. The latter situation is similar to the addition of chemical or chemimechanical pulp as a "reinforcement" to the groundwood in newsprint.

Starch or gums or other dry strength-enhancing agents are well known and may be added to the recycled fiber furnish to compensate for the loss in original strength [5].

Recycled fibers may be separated by screening into long and short fiber fractions. This permits more flexibility in the use of the secondary fiber stock by allowing the use of longer fibers where higher strength is required, such as in linerboard or in separate plies of multiply products. Equipment useful for fractionation and possible areas of application have been described [6,7].

As discussed in the chapter on bleaching (Chapter 23), the deinking solution is usually alkaline (except when wet strength resins are involved), and hydrogen peroxide may be added at that stage to prevent alkali yellowing. Both alkali and hydrogen peroxide have some positive effect on the tensile strengths of recycled mechanical pulp papers, at least in the first recycle (Fig. 1).

Treatment with ozone is also known to increase the strength properties of mechanical pulps [8]. There are no known mill applications of ozone at this time, but three commercial ozone bleaching systems are scheduled to be in operation by the end of 1992 [9]. The technology for strength enhancement by ozone treatment could thus be readily adapted from bleaching experience.

Oxygen is also reported to have a beneficial effect on the strength properties of recycled fibers [10]. Many mills employ oxygen gas to oxidize kraft black liquors, as a prebleach stage after kraft pulping or to reinforce an alkaline extraction stage during conventional bleaching sequences. Oxygen is especially useful in providing cleaning and shive removal (Chapter 23), and under some circumstances may provide some strength improvement as well.

The press section of the paper machine assists water removal from the wet web and improves interfiber bonding by increasing fiber-to-fiber contact. Various modifications have recently been proposed for the press section. One such modification, the extended press nip, permits an increase in the proportion of recycled paper in the furnish while maintaining strength properties [11]. However, if the fibers were initially poorly bonded (for example, mechanical pulps), the extended nip could not increase the strength of the recycled fibers [12].

RESEARCH ON STRENGTH ENHANCEMENT

The Forest Products Laboratory in conjunction with the American Newspaper Publisher's Association and the U.S. Environmental Protection Agency initiated a research program to recycle newsprint using a minimum amount of water. The objective was to design a process that could be used in urban regions that are close to the source of old newspapers but that are often restricted in the amount of water available for processing. The initial stages of the program did not include deinking. The research began with a dry-fiberizing stage, followed by air-forming of a new sheet and press-drying to densify and promote interfiber bonding. We recognized that if this dry recycling process were successfuly developed, an entirely new deinking concept would be necessary. A recent U.S. patent addresses this issue and provides a lead for further research and development [13].

Many methods of dry-fiberizing were studied [14]. Methods that showed some potential were hammermilling or single-disk refining. The challenge was to mill sufficiently to produce individuality separated fibers without extensive cutting and fines generation. This usually resulted in a trade-off. After exploring several processing variables, the best fiber produced retained 97% of the fiber length; wet-formed, press-dried handsheets had 93% of the tear strength, 69% of the tensile strength, and 46% of the tensile energy absorption of handsheets made from wet-fiberized fibers. When air-forming was compared with wet-forming, the best results were 53% of the tensile strength of the wet-formed papers. Methods were therefore sought to improve the interfiber bonding of the dry-fiberized recycled fiber.

Methods and Materials

The starting material was commercial newsprint supplied as rollstock and containing no print. The newsprint was composed of 75% spruce groundwood and 25% spruce sulfite pulp.

A dry-fiberizing process was chosen that readily prepared a 100% yield of individually separated fibers. However, the processing resulted in considerable fines generation and low handsheet tensile strengths. Newsprint with approximately 7'% moisture content was cut into 8- to 10-cm strips and fibrized in an 8-in. Bauer[2] disk refiner with the plate gap set at 0.125 mm. The fiber was collected in a vacuum trap on a vacuum bag filter. The fiber distribution and the tensile properties are given in Table L

For wet fiberization, the rollstock newsprint was defiberized in a Morden hydropulper with a water–to-paper ratio of 33 and water temperature of 65°C. The slurry was mixed for 25 min, then dewatered on a 200-mesh (74-µm) screen, followed by pressing to 25% solids content. The moist cake was crumbed and stored at 8°C until used.

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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.

Table I. Fiber length distribution and tensile properties of dry-fiberized newsprint[a]

Screen classification (Bauer McNett)	
Screen mesh (μm)	Percentage of total retained
595	18
297	31
149	20
74	5
<74	26

[a] Weighted average fiber length, 0.92 mm; tensile index, 7 N m/g; tensile energy absorption index, 25 J/kg. 70 g/m^2 handsheets.

Gas-phase treatments of the newsprint (10 g, ovendry basis) were conducted in laboratory rotory evaporators modified slightly to permit introduction of the gas directly into the round-bottom flask and analysis of the exit gas. For nitrogen dioxide, gas was obtained from the liquid state in an apparatus described in reference [15]. Chlorine dioxide was carried into the reaction flask by nitrogen bubbling through a solution of chlorine dioxide in cold water. Sodium hydroxide solutions (0.02 M) or ammonium hydroxide solutions were used, although ammonia gas was tested to verify that a gas-phase post-treatment could also be used. Runs at alkaline pH levels were not post-treated. The reacted fibers were then soaked in 250 ml of alkali (0.02 M NaOH unless stated otherwise). After 30 min, the pulp was washed free of alkali on filter paper and made into handsheets by TAPPI Test Method T205. Handsheets were made to a basis weight of 70 g/m^2. The handsheet tensile tests were performed according to TAPPI Test Method T494.

Sheets were air laid onto a 75-µm fluorocarbon screen in the apparatus described in reference [14]. The formed sheet and the screen were transferred to a balance on a tared brass carrier. The moisture content was adjusted as necessary in a humidification chamber supplied with ultrasonically generated water vapor. The moistened sheet was subjected to ozone in a treatment vessel consisting of a stainless steel beaker with a 200-mesh (74-µm) stainless steel screen bottom. The beaker was covered with a Teflon top fitted with a fritted glass gas sparger for introduction of gases. After ozone treatment, the sheet was treated with ammonia gas in the same apparatus. The treated sheet was dampened thoroughly with water and wet pressed to 60% moisture content. The screen was then removed and the sheet was press-dried following the procedure described in reference [14].

Results

Cause of Strength Loss

Scanning electron photomicrographs showed that in addition to considerable fiber cutting and generation of debris, the processing of wet-formed handsheets from dry-fiberized pulp produced handsheets with a distinct lack of the fibrillation needed for interfiber bonding (Fig. 2). This result was probably caused by thermal effects during the dry refining and could be horrification of the polysaccharides or flow of the thermally softened lignin.

Figure **2—Electron micrograph of wet-formed handsheet made from unbeaten dry-fiberized newsprint.**

To distinguish between the cutting and heating effects, wet-fiberized newsprint was made into handsheets with couching but no heat drying. The sheets were allowed to reach equilibrium in a conditioned test room and were then cut manually into strips 5 mm wide. These strips were then cut into pieces approximately 1 mm wide. This cutting produced a pulp with a weighted fiber length reduced from the original 1.55 mm to 0.84 mm. The cut pieces were then reslushed into pulp at room temperature and formed into handsheets by standard procedures.

The results of strength tests are shown in Figure 3. The cutting procedure reduced the tensile index from 32.6 to 28.2 N m/g for the unbeaten pulp. This was only a portion of the loss sustained by dry-fiberizing. The unbeaten dry-fiberized pulp had a very low tensile index of 6.6, but very high freeness, which reflects the lack of fibrillation. The wet-fiberized, cut fibers retained good fibrillation properties, as indicated by the scanning electron micrograph of a handsheet from the unbeaten pulp (Fig. 4). Therefore, we conclude that the thermal effect on the fibers during dry-fiberizing is a major factor in the tensile strength loss of recycled paper. Steam injection during refining or pre-equilibration of newsprint strips to 95% relative humidity improved tensile energy absorption and weighted fiber length of dry-fiberized fiber [14].

A control pulp that was made into handsheets but not cut before reslushing had a higher tensile index (38.6 N m/g) than that of handsheets made from the original wet-fiberized fiber. This may reflect the increased flexibility in mechanical fibers upon gentle recycling, as noted by Howard and Bichard (Chapter 16).

Physical Treatments

Forming handsheets from hot water rather than cold was shown to increase tensile strength [14]. The authors postulated that at least part of the mechanism might be removal of latency. However, if the dry-fiberized pulp suspension were heated to 80°C and then cooled prior to handsheet formation, the resultant tensile strength (Fig. 5) was slightly lower than that of unheated pulp. Hot water forming must improve fiber flexibility and bonding potential at the time of formation. The positive effect of hot water forming was also observed with sheets that were subsequently press-dried and with pulps that were treated with ozone prior to sheet formation.

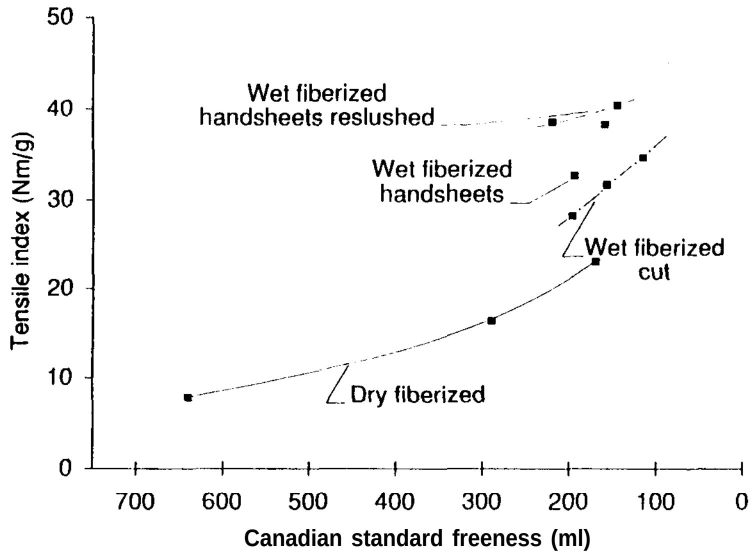

Figure 3—Effect of cutting on tensile strength of wet-fiberized fibers compared to strength of dry-fiberized newsprint (70g/m^2 handsheets).

Figure 4—Electron micrograph of wet-formed handsheet made from unbeaten, cut newsprint fibers.

One option for mills for enhancing the strength of paper made from recycled fibers is to blend the recycled pulp with virgin pulp or other long-fiber pulp fractions. In our study, dry-fiberized recycled newsprint was blended with 5% and 15% bleached Southern Pine kraft pulp. The low level of virgin long-fiber pulp was chosen because this quantity is often added to mechanical pulp in the production of first-time newsprint. Strength was improved significantly, although not to the level of the original newsprint (Table II). We implicitly assumed that the addition of longer fibers would be necessary to improve the

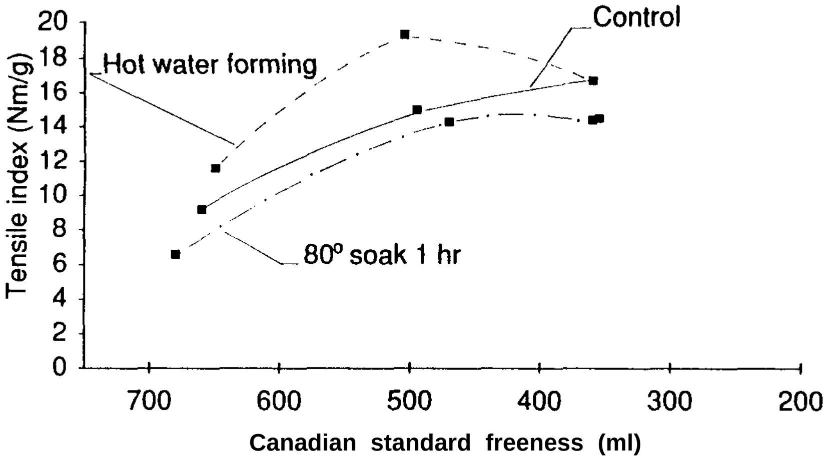

Figure 5—Effect of of hot water forming compared to *hot* water *soakng on tensile strength of 70-g/m² handsheet,*

Table II. Results of blending virgin Southern Pine with dry-fiberized newsprint[a]

Fiber	CSF[b] (ml)	Tensile index (N m/g)	TEA index[c] (J/kg)	Tear index (mN m^2/g)
Dry-fiberized newsprint	290	16.4	92	—
Dry-fiberized newsprint + 15% Southern Pine kraft	320	25.7	242	4.4
Ozone-surface-treated dry-fiberized newsprint	200	19	139	—
Ozone-treated newsprint + 15% Southern Pine kraft	260	24.8	205	3.9
Rollstock				
Machine direction	—	52.1	510	—
Cross-machine direction	—	19.6	384	—

[a] 70 g/m^2 handsheets.
[b] Canadian standard freeness.
[c] Tensile energy absorption.

tearing strengths. The higher level of virgin pulp (15%) increased tearing strength, but not to the level of wet-fiberized pulp (6.0 mN m^2/g).

Preliminary experiments were conducted on the addition of adhesives to dry-fiberized fiber [14]. Pearl starch, plasma protein, and casein were each evaluated at a level of 10% of the fiber weight. The handsheets were press-dried after formation and then tested for tensile strength (Fig. 6). Each additive improved strength; casein produced the best results.

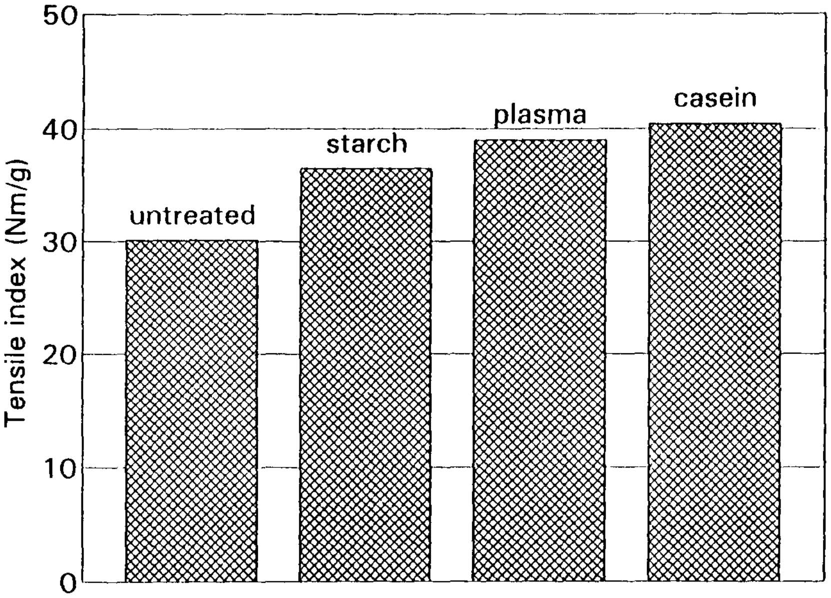

Figure 6-Increases in tensile index obtained from addition of 10% solutions of adhesives to dry-tiberized newsprint (70 *g/m^2 handsheets). All sheets were press-dried [14].*

Chemical Treatments

In keeping with the plan to use a minimum of water in the recycling process, several gas-phase chemical treatments were evaluated for their ability to enhance fiber bonding strength. Oxidative systems were considered likely to succeed because of possible surface generation of carboxyl groups and surface delignification to expose more carbohydrate hydroxyl groups. Both types of reaction have the potential to increase hydrogen bonding between fibers.

Nitrogen dioxide, chlorine dioxide, and ozone were chosen as gases likely to produce the desired result. Ozone showed the most promise under the experimental conditions (Table III). The proportion of chlorine dioxide applied was limited for safety reasons. Nitrogen dioxide resulted in dark orange pulps with very little strength enhancement.

The ozone treatment was evaluated by using a central composite experimental design. The variables were time, temperature, and moisture content. The rate of ozone application was held constant at 0.06 g/min, so the quantity applied varied with time. All treated samples were soaked in 0.02 M sodium hydroxide solution for 30 min, then washed and wet-formed into handsheets. The results are shown in Table IV. After the minimum react ion time of 10 to 15 min, the most important variable was moisture content. Lower temperatures gave better results.

Table III. Gas-phase treatments of dry-fiberized newsprint[a]

Treatment	Charge (%)	Time (min)	Temperature (°C)	Moisture content (%)	Tensile index (N m/g)
(Control)[b]					10
Nitrogen dioxide	5.4	15	45	79	11
Chlorine dioxide	0.06	90	ambient	50	12
Ozone	4.4	20	40	50	21
Ammonia	—	30	ambient	7	9
Ammonia	—	30	ambient	50	4
Ammonia + Ozone	—	30	40	50	19

[a] 70 g/m^2 handsheets. All samples post-treated with 0.02 M NaOH.
[b] Dry-fiberized post-treated newsprint.

Table IV. Central composite design for ozone treatment of dry-fiberized newsprint[a]

Time (min)	Temp. (°C)	Moisture content (%)	Run order	Tensile index (N m/g)	TEA index (J/kg)
15	35	30	17	17	89
15	35	70	9	22	148
15	45	30	19	10	44
15	45	70	10	20	121
45	35	30	7	16	73
45	35	70	2	21	90
45	55	30	16	12	49
45	55	70	6	25	152
5	45	50	15	17	81
55	45	50	18	27	173
30	28	50	5	33	248
45	62	50	20	14	71
30	45	16	12	10	36
30	45	84	13	48	443
30	45	50	4	24	140
30	45	50	1	22	129
30	45	50	14	28	175
30	45	50	3	26	165
30	45	50	11	28	188
30	45	50	8	24	155

[a] 70 g/m^2 handsheets.

An investigation of the properties of the treated fibers showed that considerable delignification occurred in those fibers that gave the strongest handsheets. A plot of tensile index as a function of lignin content shows a direct proportionality (Fig. 7). If the fibers are delignified with sodium chlorite, a similar plot is obtained, but the strength is higher at any given lignin content. This is undoubtedly a reflection of the more selective and

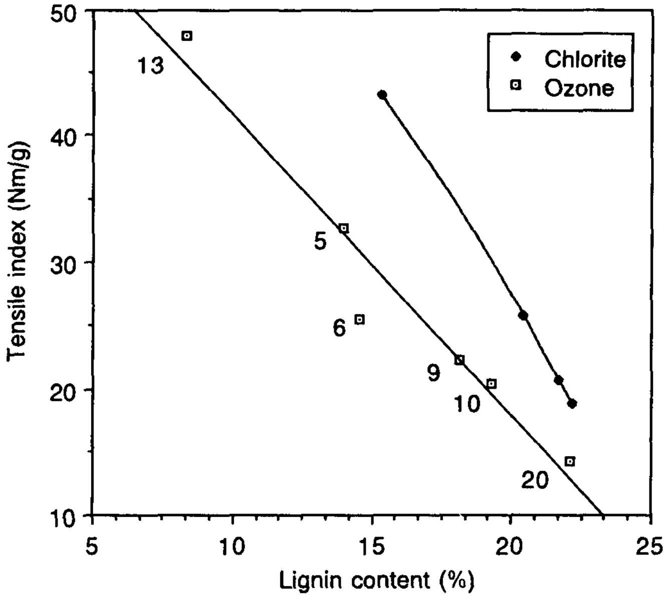

Figure 7—Tensile indexes of chlorite- and ozone-treated dry- fiberized 70-g/m^2 handsheets in relation *to* lignin content. *Numbers indicate run order in Table IV.*

milder oxidizing action of sodium chlorite and provides a basis for believing that chlorine dioxide would also bean effective gaseous reagent if the gas-phase concentration could be safely controlled.

Air-Formed Papers

Conventional recycle papermaking uses two unit processes that require a considerable amount of water. The first process is wet-fiberizing and washing (including deinking). The second is paper formation, which usually employs a very dilute suspension (ca. 0.5%) of fibers in water.

Air-formation of dry fibers into paper products is known, but the bonding strength is very low and synthetic polymers are usually added as needed to provide cohesive strength and other desired properties. Air-forming is not used for writing or printing papers. If air-formed papers were to be made suitable for such use, interfiber bonding would have to be considerably enhanced.

One physical method of enhancing interfiber bonding is to restrain the paper web during drying. The Press-Dry process, developed at the Forest Products Laboratory, has been shown to effectively increase the tensile strength of papers made from short, stiff, high-yield hardwood fibers [16]. The shortened mechanical pulp fibers obtained from dry-fiberizing should be amenable to strength enhancement by press-drying. The air-formed papers should be especially benefitted by press-drying because of the need for densification. Press-drying has not been applied commercially. For a critical review of the status of press-drying and related techniques, see reference [17].

Without some form of pressing, air-formed papers have little interfiber bonding strength. Therefore, the properties of press-dried, wet-formed sheets were compared with those of the original newsprint. The results indicated that press-drying of hot-water-formed paper increased the tensile index compared to that of the original newsprint

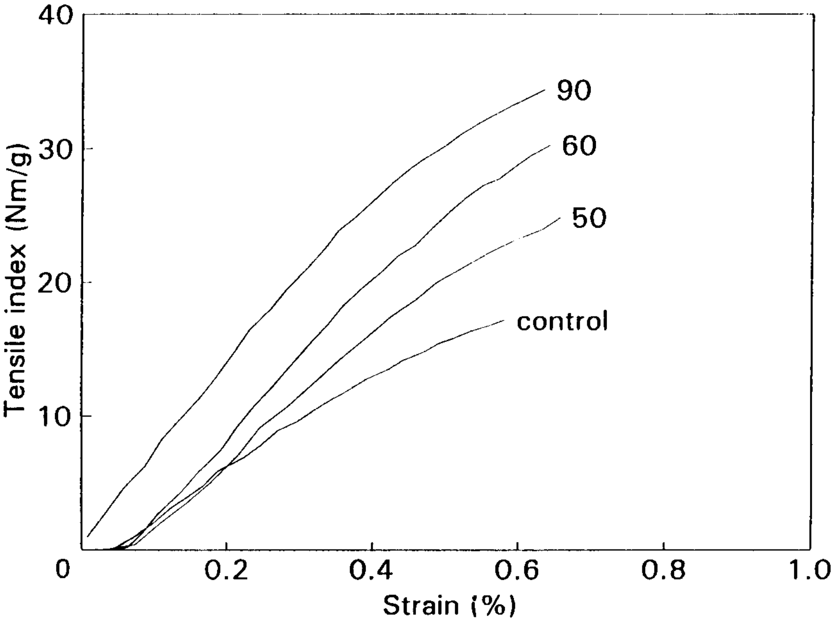

Figure 8—Stress-strain curves for dry- fiberized, air-formed 70-g/m² handsheets treated with ozone, neutralized with ammonia, and press-dried. Numbers indicate moisture content during ozone treatment.

(Fig. 7) [14]. Note that hot water rewetting of the air-formed sheets prior to press-drying gave better bonding strength than did cold-water rewetting. The strength of air-formed papers, however, did not reach that of the wet-formed papers or of the original newsprint under any conditions.

If the surface of dry-fiberized fibers were modified chemically to promote interfiber bonding, press-drying of air-formed sheets should provide the necessary fiber-fiber contact. Therefore, we investigated the ozone treatment of fibers to be air-formed and press-dried. Conceptually, it would be a simpler engineering process to treat fibers with ozone gas in a reactor and then air-form the fibers into a web that could be press-dried without further disruption. However, because the ozone treatment enhances interfiber bonding, the treated fibers tend to clump together in tight balls. We were unable to find a method for making satisfactory air-formed sheets from treated fiber.

A satisfactory procedure was developed by air-forming the sheet before ozone treatment. The air-formed sheet was moisturized to the desired moisture content and treated with moist ozone gas. After flushing the ozone with oxygen, the acids generated by the ozone treatment were neutralized with ammonia gas. The sheets were then press-dried.

The results are shown in Figure 8. The different curves are averages of the results obtained at different moisture contents during the ozone treatment. The tensile strength of the air-formed, press-dried sheets was significantly improved. However, tensile strength did not quite reach the level of the wet-formed, press-dried sheets or the level of the original newsprint (see Table V).

Table V. Summary of tensile strength tests

Fiber	Tensile index (N m/g)
Rollstock	
Machine direction	52
Cross-machine direction	20
Geometric mean	39
Press-dried wet-fiberized pulp	58
Dry-fiberized pulp	
Air-formed, hot rewet	23
Wet-formed, hot rewet	48

SUMMARY

The magnitude of the drying-induced strength loss of recycled fibers varies with the nature of the original fiber. Chemical pulps with very low lignin contents are more susceptible to tensile or bonding strength losses than are other types of pulp. Dry-fiberized newsprint pulp produces weak sheets because of shortened fibers and a loss of surface fibrillation. Shortened fibers affect mainly the tearing strength, which can be restored only by the addition of longer fibers. Interfiber bonding can be enhanced by higher moisture contents during defiberizing, hot water forming, addition of adhesives, blending with stronger fibers, press-drying, and chemical treatments. When the chemical treatments cause significant delignification, fiber flexibility is considerably improved and the strongest papers are obtained. Restricting the chemical action to the fiber surface gives some bond enhancement.

As part of an attempt to develop a newspaper recycling process that requires substantially less water than do current processes, the water-intensive repulping and paper-forming steps were replaced with dry-fiberizing, air-forming, gas-phase ozone and ammonia treatments, and press-drying. The tensile strength of the dry-recycled paper approached that of the original newsprint.

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