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IMPULSE DRYING OF NEWSPRINT

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IMPULSE DRYING OF NEWSPRINT

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ABSTRACT

Impulse drying, a process for drying paper in a long nip press with one heated surface, has been shown to yield superior newsprint properties with reduced energy requirements. The improvements are the result of the application of new dewatering and densification mechanisms to the drying process. Applied to newsprint, two impulse dryer nips could replace an entire cylinder drying system and, potentially, one press nip, while giving increases in sheet density and tensile strength of 30 to 40%. Impulse dried sheets have excellent surface appearance and properties, including reductions in surface roughness of up to one-half and increases in water absorption time by a factor of six. Energy use is reduced in comparison with cylinder drying, as up to 50% of the water is removed as liquid. Heat requirements as low as 930 kJ/kg (400 BTU/1b) water removed have been measured. Process design and development are continuing with the construction of a laboratory pilot impulse dryer at The Institute of Paper Chemistry to demonstrate continuous operation.

KEYWORDS: Drying, Pressing, Newsprint, Physical properties

INTRODUCTION

The water removal and sheet quality development performance of conventional drying sections are limited by the low intensity of the driving forces applied in today's processes. Large improvements in performance will require the use of much more intense dewatering and densification forces. Impulse drying is a new drying technology which applies intense conditions to produce superior sheet properties with high water removal rates and low energy consumption.

Impulse drying is the process of removing water from a formed and partially pressed web in a press nip with on every hot roll surface. The original process concept was suggested by Wahren (1) in the late 1970's, and has been under devellopment at The Institute of Paper Chemistry for the past five years. The early work on this high intensity drying process has been described by Ahrens and Sprague (2) and by Arenander and Wahren (3).

Impulse drying typically utilizes pressures of 0.3-7 MPa, temperatures from 150-500 C, and exposure times from 15 to 100 milliseconds. This brief exposure to intense conditions gives rise to dewatering and densification mechanisms which have not previously been applied to paper. Descriptions of these mechanisms have been presented in papers by Sprague and Burton (4), and by Burton, Sprague, and Ahrens (5). High pressure steam is generated rapidly in the surface of the sheet next to the hot roll, and the movement of the steam acts to displace liquid water from the sheet into the felt. Sheet temperatures near the hot roll approach 200 C, promoting fiber conformability and bonding. The middle of the sheet is preserved from excessive densification, as the external pressure is released before the sheet is completely heated through and dried. Vapor flashing in the middle of the sheet may restore a portion of the original sheet bulk. These processes result in a unique sheet structure allowing combinations of strength and optical properties that are difficult to achieve in conventional papermaking. In addition, the rates of water removal are very high, with up to half of the water leaving as liquid.

An overview of the performance of several commercially important paper grades after impulse drying has been presented by Sprague (6). The purpose of this paper is to present detailed information on the response of newsprint to impulse drying.

EXPERIMENTAL

Equipment

The equipment used in this study was a benchscale impulse drying simulator, consisting of an electrohydraulic press (MTS) with one pressing surface electrically heated to an elevated temperature. This unit is shown in Figure 1. The electronic control of the pressure-time profile can produce a variety of pressure pulses ranging from the typical bell-shaped profile of roll presses to the nearly rectangular pulse of some long nip presses. A load cell above the upper platen is used to control and measure the total load and pressure profile. The heated platen temperature is maintained using a surface thermocouple signal to control power input to the electrical resistance heaters imbedded in the platen. A ring presteamer can be added to the system to preheat the sheet prior to impulse drying.

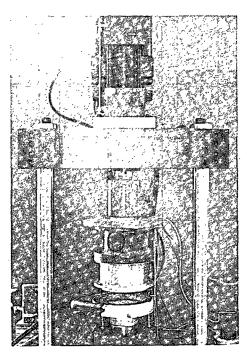


Figure 1. MTS electrohydraulic press simulator with impulse drying head installed.

Handsheets

Handsheets were made according to TAPPI standard procedures, except that the sheet diameter has been reduced to five inches to permit high pressure drying within the dynamic force limits of the MTS system. Sheets were couched and lightly pressed to a high moisture content and stored in sealed plastic bags in cold conditions. Storage time is limited to two days. Just prior to drying, the sheets were prepressed to the desired moisture content in a laboratory roll press using press impulse levels typical of commercial equipment. This pressing procedure deviates from the standard, but is used to avoid excessive densification in the pressing step.

Impulse drying

After prepressing, each sheet was weighed and then placed on a dry, mechanically conditioned wet press felt on the lower platen. The upper platen was then lowered under the control of the MTS system electronics to deliver the desired pressure pulse. In most cases impulse drying was used to remove only a part of the water originally in the sheet. Thus, immediately after impulse drying, the sheet was again weighed and then dried to approximately six percent moisture content between a dryer felt and a heated flat plate under conventional dryer conditions of about 3.5 kPa pressure and 115°C temperature. The time required to reach the desired moisture target is determined by precalibration.

Once the sheets were dried, they were conditioned according to TAPPI standards and subjected to a variety of physical property tests. The remnants from the tests were oven dried to get the dry weight needed for water removal calculations.

Control sheets for reference purposes were obtained by diverting a few handsheets from the impulse drying step and drying them entirely on the conventional drying simulator described above.

A flow chart summarizing the experimental test procedure is presented as Figure 2.

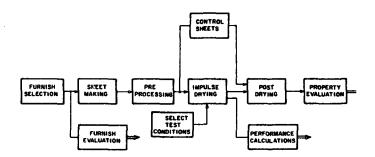


Figure 2. Elements of performance evaluation.

Furnish

The furnish used for the newsprint studies was formulated from 80% stone groundwood and 20% never-dried bleached softwood kraft. The specific surface of this mixture was 29,100 cm²/g, and the fiber specific volume was 3.38 cm³/g. The freeness of the furnish was 175 mL CSF, with a water retention value of 3.03. The handsheets were made to a grammage of 50 g/m².

Energy use measurements

The heat required to impulse dry the newsprint sheets was determined using a lithium chloride tracer technique developed by Devlin (7). A flowsheet for this technique is presented in Figure 3. The method involves saturating the adsorption sites on the fibers with aluminum nitrate, then forming handsheets using a dilute aqueous solution of aluminum nitrate and lithium chloride. The sheets were then impulse dried as described above. The lithium chloride which was carried from the sheet into the felt by the flow of liquid water was recovered by extracting the felt in boiling water and analyzing the extract for its lithium content by flame emission spectroscopy. The liquid water flow to the felt can be calculated knowing the initial concentration of lithium in the handsheet. The impulse drying heat requirements can then be calculated from a simple energy balance. The sheets formed with lithium chloride were used only for water removal rate

and energy use measurements; all physical property work was done on standard handsheets without added salts.

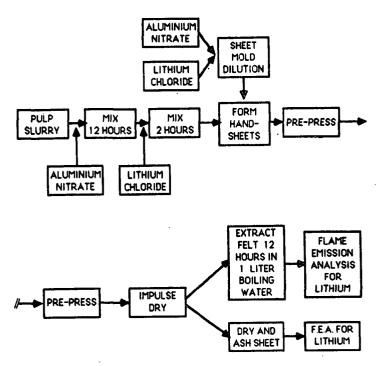


Figure 3. Flowchart for the lithium chloride displacement method for measuring liquid water removal.

RESULTS

Water removal rates

It is desirable from a machine capital cost standpoint to maximize water removal rates to keep equipment sizes as small as possible. Impulse drying performs very well against this criterion, as shown in Figure 4. The observed water removal rates of several thousand kg/h/m² are between 100 and 1000 times greater than the 25 kg/h/m² typical of cylinder drying. For newsprint, this means that the sheet can be dried from 50% solids to 85 to 90% solids in 20 to 25 milliseconds. If this amount of drying were to be done in two impulse dryer nips, each nip would need to be six to ten inches long. This is well within the nip width capabilities of existing long-nip press technology.

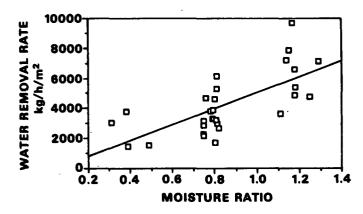


Figure 4. Water removal rate for newsprint.

Water removal rate is a strong function of ingoing moisture ratio (grams of water per gram fiber); the apparent scatter in Figure 4 is the effect of the other experimental variables of pressure, temperature, and nip residence time. A multiple regression correlation for water removal rate is presented in Table 1 and may be used to judge the effects of the other variables. The rapid, linear increase in water removal rate with increasing sheet moisture ratio means that the final moisture content leaving the impulse dryers will be similar, whatever the entering moisture content may have been. The weak dependence of outgoing moisture content on ingoing moisture content means that impulse drying offers the same degree of moisture profile levelling as wet pressing.

V = A + B*T(°C) + C*P(KPa) + D*NRT(SEC) + E*MR

V	A	B	C	D	E	R ²
WRR kg/h/m ²	-1940	13.8	0.0284	-147500	4880	.96
HEAT USE tJ/m ²	-520	0.131	0.00383	197	48.7	.91

Table 1 Multiple regression correlations for water removal rate and energy use. T = Temperature, °C, P = peak pressure in nip, kPa, NRT = nip residence time, seconds, MR = moisture ratio, kg water/kg dry fiber. All coefficient are significant at the 95% confidence level.

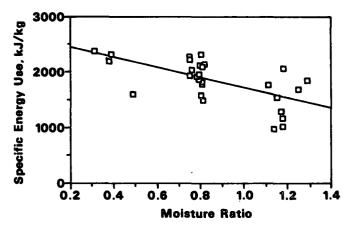
The data in Figure 4 also show that impulse drying from one side of the sheet does not diminish the ability to dewater from the opposite side. All data below a moisture ratio of 0.7 in Figure 4 relate to drying the second side of the sheet; the water had to leave through the surface previously densified in the first nip. The dependency on moisture ratio remains linear, which suggests that the densified surface is not a severe obstacle to water removal by impulse drying.

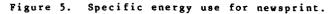
Water removal rate is also a strong function of initial sheet temperature, as a cold sheet delays the initiation of the impulse drying mechanisms and makes poor use of the available nip residence time. All data presented in Figure 4 were taken on sheets presteamed to 80°C. Room-temperature sheets (25°C) would have water removal rates about two-thirds of those reported here.

Energy consumption

The total pressure driving forces developed in impulse drying are sufficient to remove a substantial amount of the water as liquid. Liquid water removal is about 40 to 50 percent of the total water removal under many conditions. The total energy consumption of the process involves the sensible heat of the fiber and water, plus the latent heat of vaporization of that portion of the water which is evaporated. Presteaming the sheet serves to reduce the energy cost for water removal in impulse drying, since less high grade energy is consumed in raising the sheet temperature in the nip.

Specific energy consumption in the impulse dryer nip (kJ/kg) is also a strong function of initial moisture ratio, as shown in Figure 5. Energy requirements are about half the minimum 2400 kJ/kg required for evaporation of water. The decline of energy requirements with moisture ratio indicates that impulse drying may be economically effective in the third press position, as well as replacing conventional cylinder dryers. Pressure, temperature and nip residence time have weaker but significant effects on energy demand; a multiple regression correlation for energy requirements in the units kJ/m^2 of paper produced is presented in Table 1. The excellent energy use performance of impulse drying is expected to offset the increased cost of high grade energy to drive the process.





Development of Properties

Impulse drying results in high average density values, although the z-direction density is nonuniform. For newsprint, increases in average density of up to 50% in a single nip have been achieved, as shown in Figure 6. Further impulse drying can raise the density to 0.9 g/cm^3 or above. All densities cited in this report are IPC densities obtained on the IPC rubber platen caliper gage (8). These densities are slightly higher than standard TAPPI densities, but are used in the belief that they more accurately report the structural properties of the sheets. At any given initial moisture content, densification is a strong function of thermal impulse, or temperature times nip residence time, with pressure having a lesser but still significant role. Low pressures (below 3 MPa) limit the extent of densification possible with a given thermal impulse.

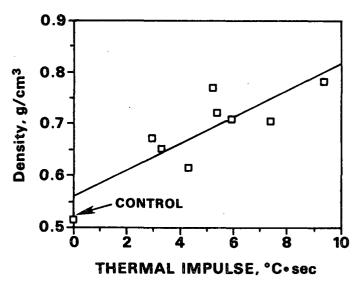
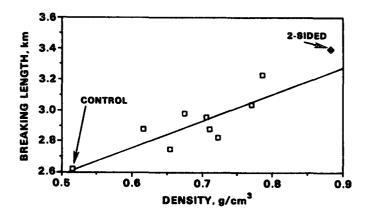
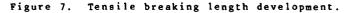


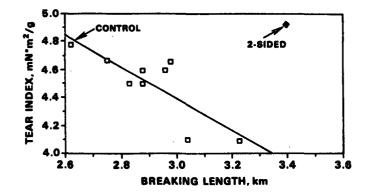
Figure 6. Density development for newsprint. This density development translates into strength improvements, as would be expected. Tensile strength increases linearly with density, whether the density is developed through one-sided or two-sided impulse drying. The total improvement in tensile strength approaches 40%, as may be seen in Figure 7. The opportunity for substantial cost savings through impulse drying lower cost furnishes to equivalent product strength is evident.

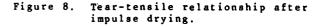




The tear-tensile relationship for newsprint can also be influenced by impulse drying. Twosided impulse drying in particular appears to allow 50% increases in tensile strength without detriment to tear strength, as may be seen in Figure 8. This potential for customizing combinations of properties is a major advantage of impulse drying and offers supstantial opportunities for product development.

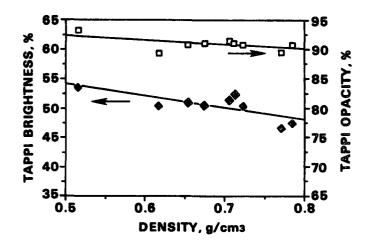
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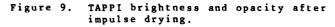




Optical properties

The high levels of densification achieved in impulse drying involve only a small penalty in sheet opacity. The sheet is much more opaque than could be expected from sheets uniformly densified to an equivalent density level. Figure 9 shows that about 4 to 5 percentage points of TAPPI opacity are lost at high levels of densification. The brightness loss is small, in the five percentage point range. Furnish substitutions to restore bulk to normal levels would probably minimize the loss in optical properties.





Other properties

1

Impulse drying also has significant effects on a number of properties which are expected to influence the conversion and printability of the product. Surface roughness (PPS) is reduced by 40 to 60 percent (Figure 10) on the side of the sheet against the heated surface. Felt side roughness tends to remain constant or increase somewhat due to felt imprinting. For two-sided impulse drying, smoothness is improved on both sides of the sheet. Calendering would still be required to bring the sheet to acceptable smoothness levels.

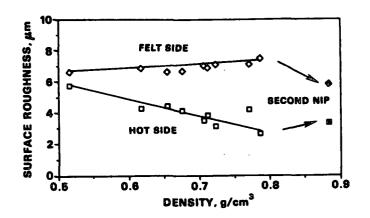


Figure 10. Surface roughness after impulse drying.

Absorbency and air permeability (Figure 11) are both reduced substantially, and pick resistance is increased. The dense surface of the sheet should be very resistant to dusting and linting.

Conversion issues such as printing quality and runnability require larger samples than those available to date. To provide such samples, a pilot roll impulse dryer is being built at The Institute of Paper Chemistry.

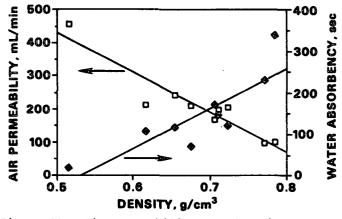


Figure 11. Air permeability (Bendtsen) and water absorbency (TAPPI T-492).

This pilot equipment will be used to produce the database of semicontinuous operation and small-scale conversion necessary for further commercial development of the process. The pilot dryer will treat one-foot wide sheets (made on the IPC web former); the impulse dryer rolls are 0.6 meter in diameter and 0.6 meter in width. The hot rolls are heated by high-temperature, electrical infrared heaters. Initial operation will be at low speeds (100 m/min), but nip conditions will accurately simulate the exposure times in high speed operation.

CONCLUSIONS

Impulse drying has dramatic and generally beneficial effects when applied to newsprint. High dewatering rates, 100 to 1000 times those found in cylinder dryers, offer the potential for

5

equipment size reduction and capital savings or increased throughput on existing dryer-limited machines. Low energy use levels, half those of cylinder drying, will offset the cost of the high-grade energy required to achieve the high temperatures and high rates of heat transfer characteristic of impulse drying.

The substantial increases in density and tensile strength may offer newsprint manufacturers the opportunity to make equivalent product using lower cost furnishes, or to improve machine runnability and production potential. The improved smoothness of impulse dried sheets may offer market advantages and product development opportunities, as well as reducing the need for calendering.

3

Impulse drying is based on pressing and heat transfer technologies which are commercially available. The high temperature of the heated roll raises a few new engineering questions, but none of these appear to pose a severe obstacle to process development. Thus, it appears that impulse drying may become a practical process with the potential for substantial improving the performance of newsprint and newsprint machines.

The remaining development issues of pilot scale operation and the processing of samples

large enough for conversion testing are currently being addressed at The Institute of Paper Chemistry.

REFERENCES

- WAHREN, D., U.S. Patent. No. 4,324,613 (April 13, 1982)
- AHRENS, F., SPRAGUE, C., High-intensity drying processes, Report 1 for Department of Energy Contract FG02-85CE40738
- 3. ARENANDER, S., and WAHREN, D., <u>Tappi</u> 66(9): 123 (1983)
- 4. SPRAGUE, C.H., and BURTON, S., Proceedings of International Conference-New Technologies in Web Consolidation and Drying, May 19-23, 1986
- BURTON, S., SPRAGUE, C.H., AHRENS, F., Fifth International Drying Symposium (IDS '86), August 13-16, 1986
- SPRAGUE, C.H., Proceedings of International Conference-New Technologies in Web Consolidation and Drying, May 19-23, 1986
- 7. DEVLIN, C., Ph.D. Thesis, The Institute of Paper Chemistry, June 1986.
- WINK, W.A., and BAUM, G.A., <u>Tappi</u> 66(9): 131-133 (1983)

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6

The work reported in this paper was supported by the members of The Institute of Paper Chemistry and by the U.S. Department of Energy. Excellent experimental help was provided by James Loughran and Lester Nett. All of these sources of support and help are gratefully acknowledged. To determine whether the ASA hydrolysis product was a sizing agent, sheets were formed with the hydrolysis product, as shown in Table 1, in a manner similar to that used with sheets containing ASA. DMVPB or alum was also added. Retention of the hydrolysis product, as measured by its IR peak, was poor relative to that of ASA, and the sheets displayed no sizing using 1% formic acid ink and a 65% reflectance endpoint. To increase hydrolysis product retention, sheets were formed at higher consistencies (1-2%), using 1-43% hydrolysis product addition levels. Sheets were made using 1) only the ASA hydrolysis product or 2) the ASA hydrolysis product and 0.15% DMVPB. Retention was improved, as the IR peak of the hydrolysis product was quite evident. These "high consistency" sheets also displayed no sizing using 1% formic acid ink and a 65% reflectance endpoint. Sheets formed at similar high consistencies with 0.25% ASA displayed strong sizing (800 seconds) using 10% formic acid ink and a 90% reflectance endpoint.

(Table 1 here)

Sheets were also formed with 0.5% ASA, stored in plastic wrap one week, and then pressed and dried. These sheets displayed significantly lower sizing levels (70 seconds using 1% formic acid ink and a 65% reflectance endpoint) than sheets formed with ASA that were pressed and dried immediately after formation (800+ seconds using 10% formic acid ink and a 90% reflectance endpoint). Delaying the pressing and drying of a sheet should yield increased hydrolysis product levels.

The above results suggest the ester formed in the ASA system is the main promoter of sizing, rather than the ASA hydrolysis product.

ASA reaction in the sheet

The ASA reaction in the sheet was examined by forming sheets with greater than usual levels of ASA (1.5%) to make the results visible by spectroscopy. Sheets

-5-

were then examined by diffuse reflectance IR for relevant peaks and their variation with conditioning, drying time, and drying temperature. Subtractive methods were used. This yielded a semiquantitative measurement of the materials contained in the sheet.

A sequence of events was found to occur within one week of sheet formation. The extent to which each event occurs and the rate at which it occurs is dependent on the drying temperature, the drying time, and the time of examination. The sequence is: 1) during drying, ASA is consumed to form esters with cellulose or the hydrolysis product with water; the ASA may also volatilize off the sheet, 2) during conditioning, more ASA is consumed, and additional esters and hydrolysis product are formed, and 3) after one week of conditioning, the ASA is fully consumed (as measured by IR examination of the sheet); the acid peak decreases and the ester peak increases relative to the levels after one day of conditioning.

In Fig. 8, ester development with drying time at several drying temperatures is shown. Increases in temperature lead to faster ester development. Ester formation appears to stabilize after about 60 minutes at 105°C, and after longer times at the lower temperatures.

(Fig. 8 here)

Figure 9 suggests that excess ASA may volatilize during drying. The products (ester and acid) appear to reach a maximum after 60 minutes, but the ASA continues to decrease after that time.

(Fig. 9 here)

Sheets extracted with toluene immediately after drying displayed decreases in the ester-acid peak. A variation in the acid or ester will cause a variation

-6-

in the other because they are mutual shoulders. Since the ester would not be extractable, the decrease must be due to hydrolysis product removal. This suggests the hydrolysis product is formed with the ester during drying.

In Fig. 10, sheets conditioned for one day show increases in the ester and the acid compared with unconditioned sheets. The greatest increase after one day is in the acid peak, suggesting that ASA remaining after drying readily yields the hydrolysis product. This may be concluded, since the acid peak becomes noticeably larger than the ester peak during conditioning at the shorter drying times - a reverse of the situation in the unconditioned sheets. At shorter drying times there would be more residual ASA present than at longer drying times. Additionally, the shorter drying times display the greatest acid increases after conditioning for one day.

(Fig. 10 here)

After one week of conditioning the acid peak has decreased, as shown in Fig. 10. This also causes the ester measurement to decrease because the acid and ester peaks are mutual shoulders. The decrease in the acid peak may be due to vaporization, but a peak simultaneously forming at about 1576 cm⁻¹ suggests the acid is involved in an unexpected side reaction. Additionally, sheets extracted with toluene after one day of conditioning, when the hydrolysis product level is still high, do not display the peak development at 1576 cm⁻¹. Extraction of the sheets with toluene after several months and IR examination of the extract yielded definite evidence that the hydrolysis product was still present and that some unreacted ASA remained.

In Fig. 11, IR examination of sheets extracted with toluene to remove the hydrolysis product shows that the ester also continues to form after drying.

-7-

(Fig. 11 here)

Conclusions

ASA clearly forms esters with cellulose and promotes sizing primarily by esterification. The hydrolysis product is a poor sizing agent and could be, as suggested by others, a desizing agent (<u>13</u>). The rate of sizing development is dependent on the drying temperature, and the extent of sizing is dependent on the conditioning time, the drying time, and the drying temperature. Long term drying of the sheet causes sizing to decrease. Conditioning of the sheets, when unreacted ASA would be expected to be present, yields increased sizing levels.

The rate of ester formation is dependent on the drying temperature, and ester formation continues after drying. Hydrolysis of the ASA occurs during drying, and extensively after drying if ASA is available. The acid groups increased during initial conditioning and then decreased after one week. Unexpected side reactions of an undetermined nature appeared to consume the acid, but loss of the hydrolysis product through vaporization cannot be eliminated from consideration. Finally, after several months, unreacted ASA and the hydrolysis product were still present in the sheet.

Experimental procedures

The pulp used was a bleached, commercial cotton linters. The pulp was treated in a Valley Beater to 290 mL CSF and then Soxhlet extracted in 1:1 chloroform: 95% ethanol for 48 hours, washed in distilled water, air dried, and bagged.

The polymer, DMVPB, was synthesized by Eggert. The approximate weight average molecular weight is 10^6 (14).

-8-

The ASA was determined by GC/MS to be a mixture of isomers, primarily of a molecular weight of 322 g/mole. Silation of the hydrolysis product was used to obtain volatility.

-9-

The hydrolysis product was prepared by reacting ASA with a small excess of distilled water at room temperature for 3 days.

The reaction of ASA in N,N-dimethylformamide was performed at 85°C for 1.25 hours. Approximately 3.5 grams of cotton linters was mixed with 100 mL of N,N-dimethylformamide, 25 mL of ASA, and 0.3 mL of triethylamine. The reacted linters were filtered, Soxhlet extracted in toluene, and oven dried to remove residual N,N-dimethylformamide.

Emulsions of ASA or the hydrolysis product were made using distilled water in an Eberbach 8590 mixer. ASA emulsions were prepared at 1.5% ASA and used immediately to prevent hydrolysis. A new emulsion was used for each sheet.

Handsheets (1.8 0.D. grams) were made using prewetted cotton linters dispersed in a British Disintegrator. The pulp was diluted to the proper consistency (generally 1%) and adjusted to a pH of 7.5. The ASA or hydrolysis product emulsion was gently stirred with the pulp two minutes, and 0.15% DMVPB was added immediately afterward with gentle stirring for a further two minutes. The pulp was added to a British handsheet mold filled with deionized water that had been preadjusted to a pH of 7.5. Sheets were formed and pressed in accordance with TAPPI Standard T205 om-81 and oven dried in a convection oven immediately to prevent hydrolysis of the ASA. All sheets containing either ASA or the hydrolysis product were dried one hour at 105°C, unless otherwise noted. Sodium hydroxide and sulfuric acid were used for pH adjustment. Samples were conditioned at 72°F and 50% RH, unless otherwise noted. Extraction of sheets was performed using a Soxhlet extraction apparatus with toluene for 14-18 hours.

Size tests were performed using a Hercules Sizing Tester, 10% formic acid ink, and an 80% reflectance endpoint, unless otherwise noted. Test ink was applied to the felt side of the sheet.

IR spectra were produced using a Nicolet 7199 FT-IR at 2 cm⁻¹ resolution. Diffuse reflectance spectra were obtained using 500 scans in a Harrick's Praying Mantis unit mounted in the FT-IR. A potassium chloride background was used for all samples. All spectra were base line corrected from 400-4000 cm⁻¹.

Subtractive results were obtained using a cotton linters sheet and a $1350-2000 \text{ cm}^{-1}$ subtraction range. Results were base line corrected from $1685-1900 \text{ cm}^{-1}$ before measuring the peak height.

Transmission spectra of extracts were obtained on sodium chloride plates using the instrument described above.

Literature cited

- 1. Dumas, D. H., Tappi J. 64(1):43(1981).
- 2. Davison, R. W., Tappi J. 58(3):48(1975).
- 3. Wingrove, A. S., Caret, R. L., Organic Chemistry, 1st edn., Harper and Row, New York, 1981, p. 985.
- 4. Fessenden, R. J., Fessenden, J. S., Organic Chemistry, 1st edn., Willard Grant Press, Boston, 1979, pp. 622-623.
- 5. Merz, J., Rohringer, P., Bernheim, M., Das Papier 39(5):214(1985).
- 6. Pisa, L., Murckova, E., Papir Celuloza 36(2):V15(1981).
- 7. Roberts, J. C., Garner, D. N., Cell. Chem. Technol. 18:275(1984).
- 8. Rohringer, P., Bernheim, M., Werthemann, D. P., Tappi J. 68(1):83(1985).

-10-

- 9. Odberg, L., Lindstrom, T., 1985 Intl. Sym. on Wood and Pulping Chem.-Tech. Papers, Canadian Pulp and Paper Association, p. 41.
- 10. Lindstrom, T., 1986 Papermakers Conf. TAPPI Press, Atlanta, p. 29.
- 11. Nahm, S., J. Wood Chem. Tech. 6(1):89(1986).
- Silverstein, R. M., Bassler, G. C., Morril, T. C., Spectrometric Identification of Organic Compounds, 4th edn., John Wiley and Sons, New York, 1981, p. 122.
- 13. Wasser, R. B., 1985 Alkaline Papermaking Conf. TAPPI Press, Atlanta, p. 17.
- 14. Eggert, A. R., The Role of Particle Size and Molecular Weight on the Adsorption and Flocculation of Polystyrene Latex with Poly(1,2-Dimethyl-5-Vinylpyridinium Bromide), Ph.D. thesis, The Institute of Paper Chemistry, Appleton, Wisconsin, 1976.

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Portions of this work were used by (WRM) as partial fulfillment of the requirements for the Ph.D. degree at The Institute of Paper Chemistry.

%ASA Hydrolysis Product	<u>% DMVPB</u>	<u>%Alum</u>	Hercules Size Test (secs.)
0.5 ¹	0	0.7	0
0.5 ¹	0.15	0	0
1.0 ¹	0.15	0	0
0.5 ²	0	0.35	. 0
0.5 ²	Ο΄	0.7	0
0.5 ²	0.15	0	0
1.0 ²	0.15	0	0

Table 1: Compositions of sheets containing the hydrolysis product and
their respective size tests.

¹Hydrolysis product added to pulp before DMVPB or alum. ²Hydrolysis product added to pulp after DMVPB or alum.

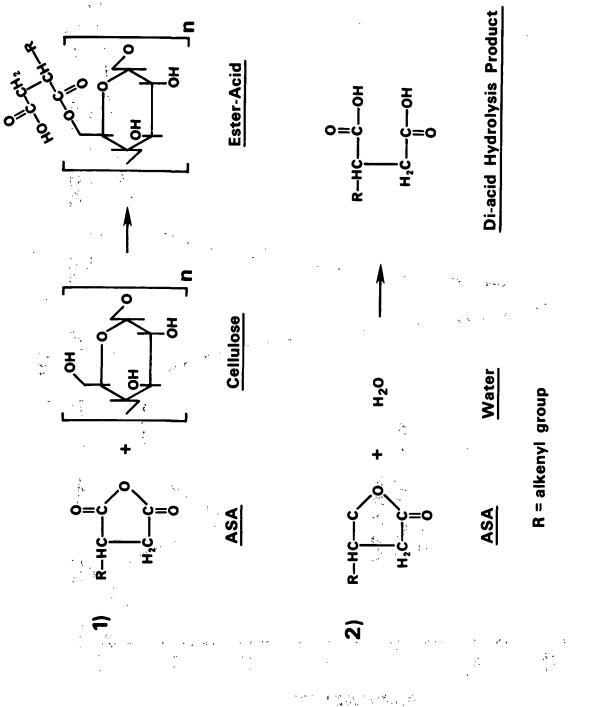
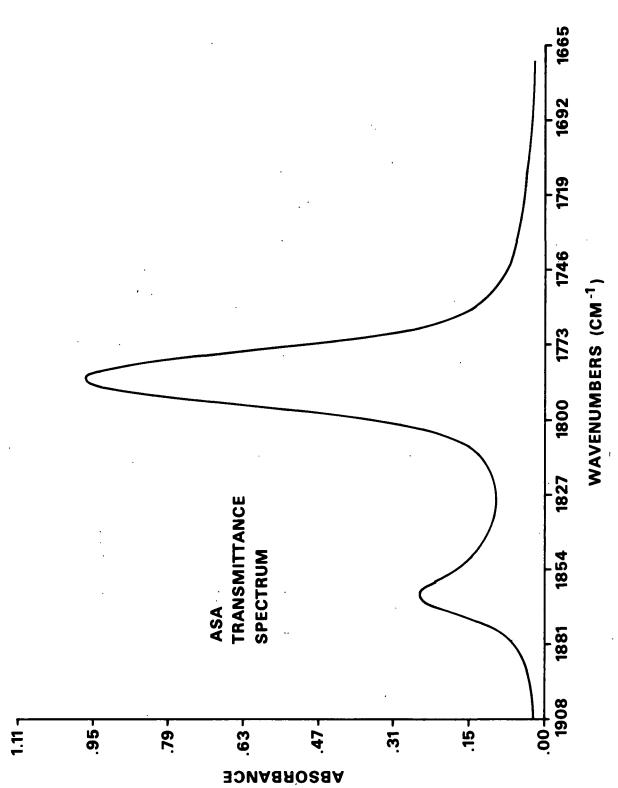
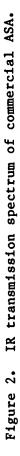
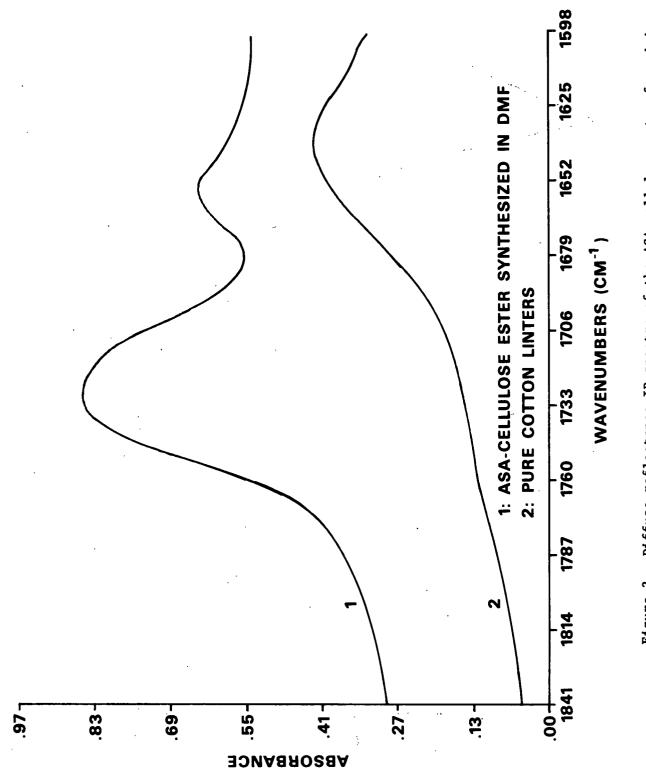


Figure 1. ASA reactions in a papermaking system.

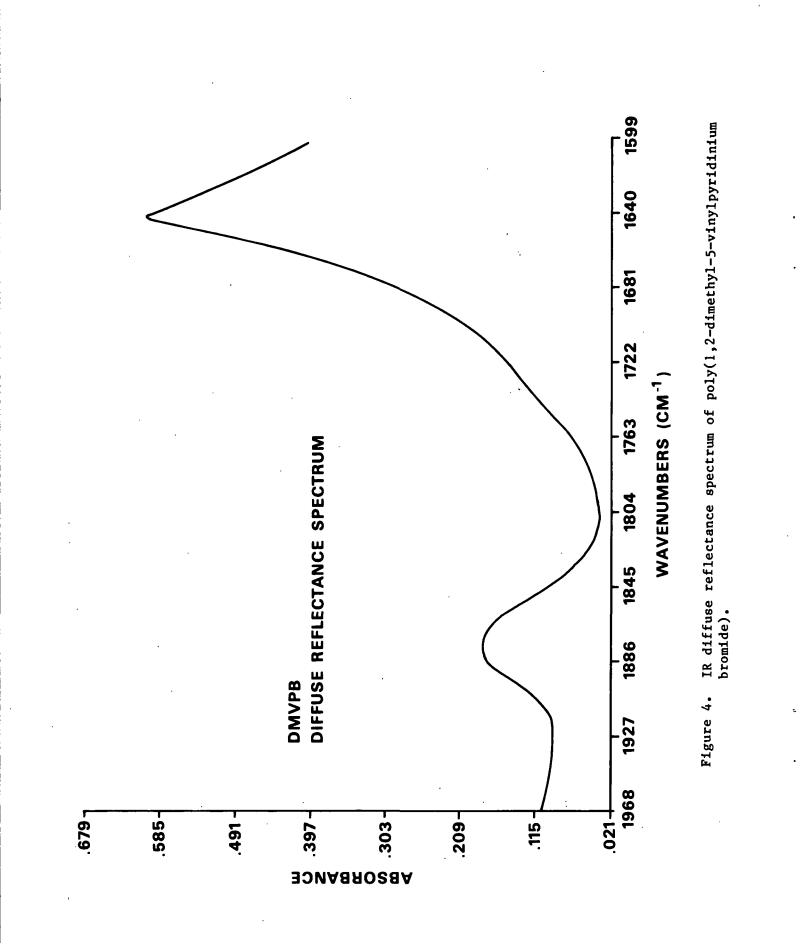


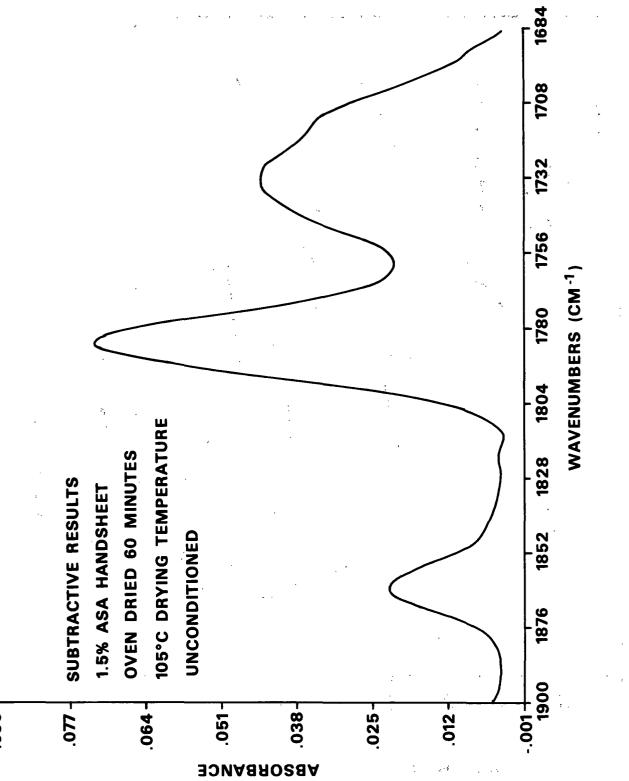




Diffuse reflectance IR spectra of the ASA-cellulose ester formed in DMF, and pure cotton linters before reaction. Figure 3.

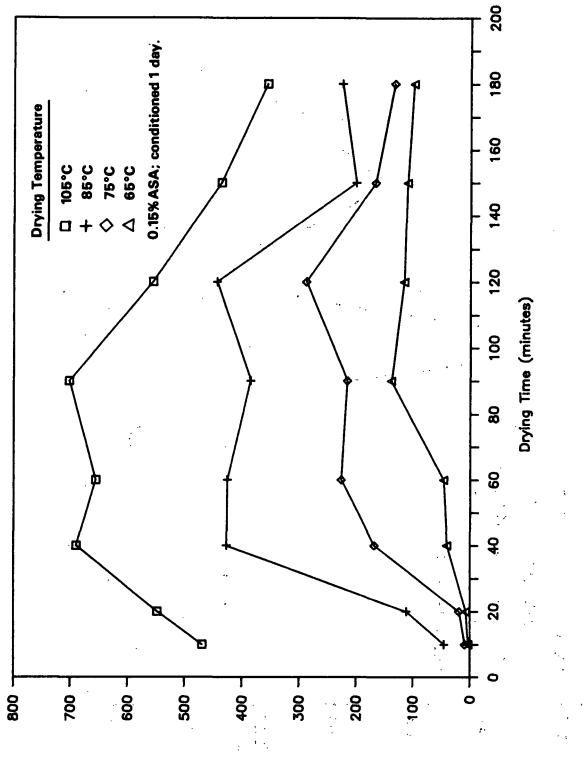
F1g





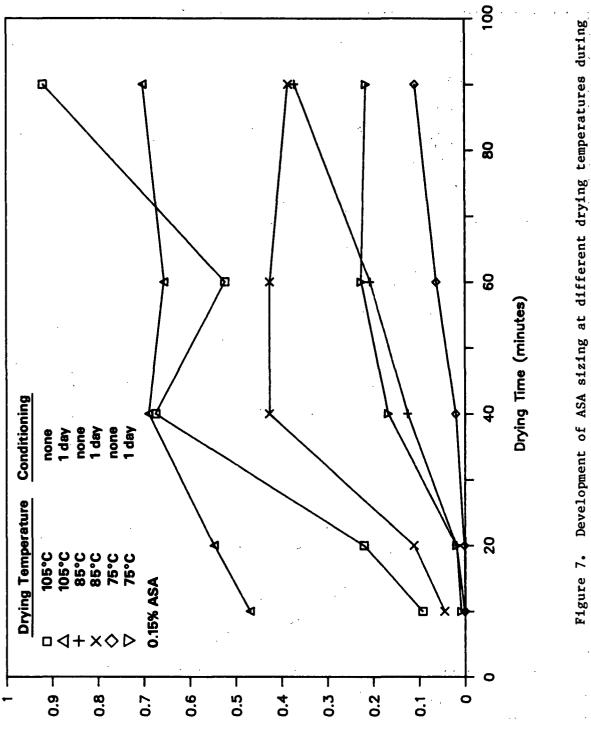


L060.



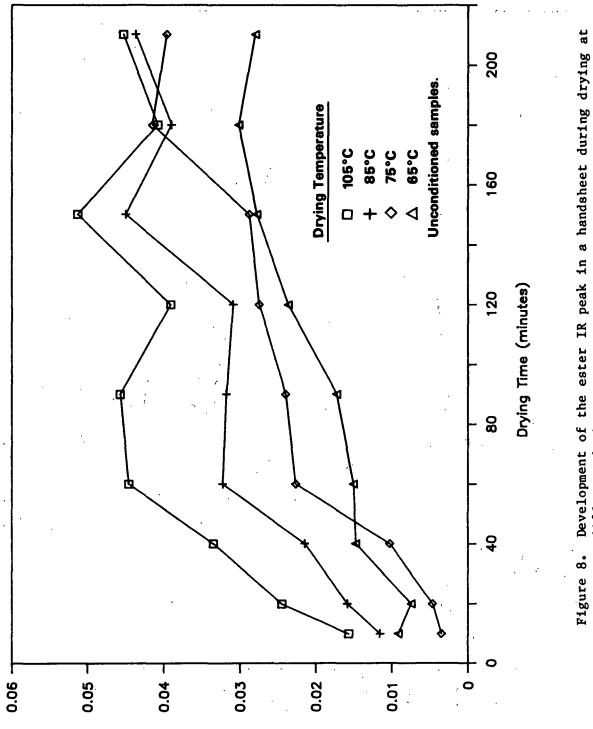


Hercules Size Test (seconds) *



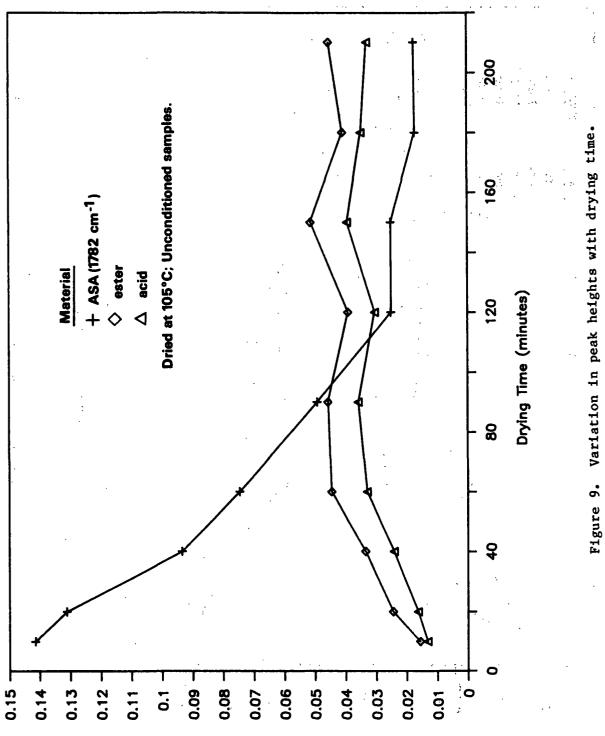
Hercules Size Test (seconds) (Thousands)

conditioning.



different drying temperatures.

R peak height (absorbance units)



R peak height (absorbance units)

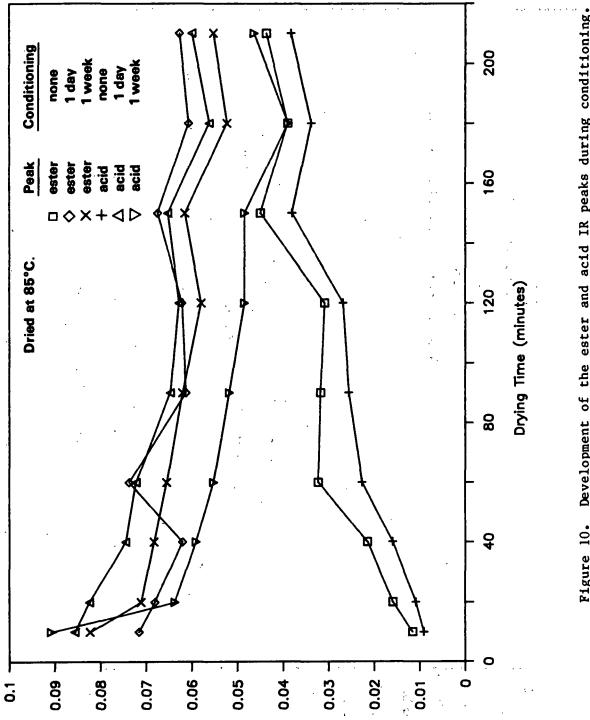
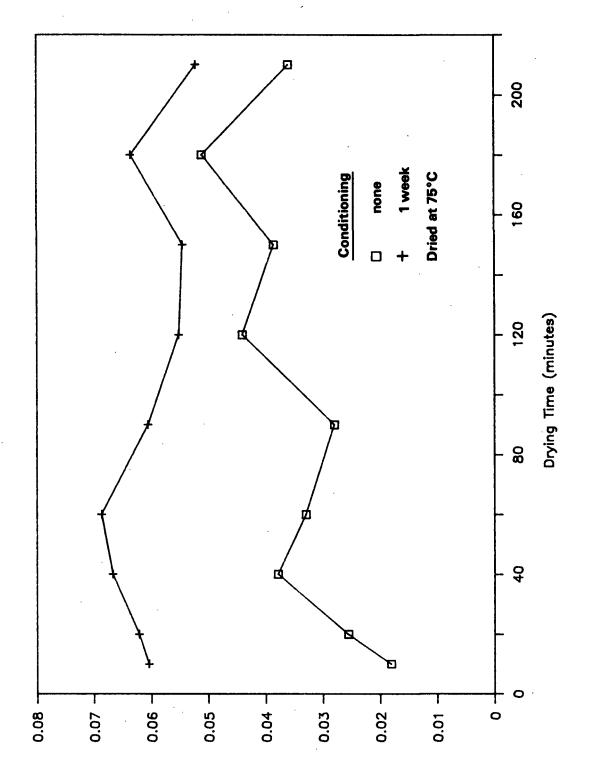


Figure 10. Development of the ester and acid IR peaks during conditioning.

ik peak height (absorbance units)



Development of the ester IR peak during conditioning. The sheet was extracted to remove the hydrolysis product after conditioning. Figure 11.

IR peak height (absorbance units)