Cellulose/polypropylene composites: the use of AKD and ASA sizes as compatibilizers

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Abstract

Alkylketene dimer (AKD) and alkenyl succinic anhydride (ASA) were used as compatibilizers in both solution and emulsion forms to treat 100 percent cellulose and 70/30 wt.% cellulose/polypropylene composite sheets made by both an air-formation technique and traditional wet handsheet formation. In all cases, the air-formed sheets had poorer mechanical properties than did the handsheets. While the treatments appeared to have a visible effect on the spreading of polypropylene over cellulose, the effects of the treatments on mechanical properties was almost always negative. Annealing the wet-formed handsheets for 5 minutes at 130°C before pressing gave control sheets with improved modulus, strain-to-break, and burst strength, but had a slight negative effect on air-formed sheet properties. The effect of treatments on the water durability of the air-formed sheets was minimal or adverse, while for the wet-laid handsheets, the treatments were slightly beneficial if the sheets had been annealed, but detrimental if they had not.

Introduction

It has been widely noted that a key to good composite properties lies incompatibility between matrix and filler. In the wood fiber-polyolefin system, the highly polar nature of the cellulosic surface does not lend itself to strong interactions with the non-polar polymer. Modification of the cellulose surface, either by chemical coupling agents or grafting, has proven to be beneficial in improving the strength and modulus, as well as the water resistance of these composites (4,7,8,11,12,14,15,17,19).

In the realm of paper manufacturing, sizing agents are one of the most widely used methods of changing the hydrophilic nature of cellulose. In particular, alkyl ketene dimer (AKD) and alkenyl succinic anhydride (ASA) are two of the most common sizes used in alkaline papermaking (Fig. 1) (2,3). The intriguing feature of these sizes is the dual polarity of their structure a polar component to react with the cellulose surface and a non-polar (hydrophobic) component which should be compatible with polyolefins.

The purpose of this study was to discover what effect treatment of cellulose pulp with AKD and ASA, in both solution and emulsion form, had on the mechanical properties of cellulose and polypropylene composite sheets made by both air-forming and conventional wet handsheet forming.

Experimental

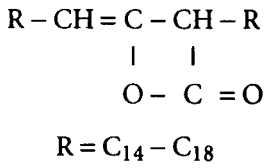
Materials

Bleached kraft cellulose pulp in dry-lap form (Ultranier-J) was obtained from ITT Rayonier. Before use, the pulp was slurried, dewatered, and fiberized (Bauer single-disk mill refiner, 3,600 rpm, 0.005-in. disk separation).

The polypropylene used was Hercules Pulpex P AD-H, which contains a surface treatment of wt.% poly(vinyl alcohol) for better water dispersibility.

Hereon 70 (Hercules) AKD emulsion was diluted to prepare the AKD emulsions used in the study. Aquapel 364, a

Alkyl ketene dimer (AKD)



$R = C_{14} - C_{18}$

Alkenyl succinic anhydride (ASA)

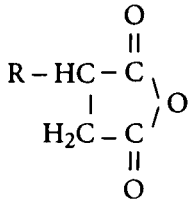


Figure 1. — Chemical structures of AKD and ASA.

dry AKD wax, also provided by Hercules, was used in all AKD/toluene solutions.

ASA/toluene solutions were made using Accosize 18 (American Cyanamid) synthetic size, while ASA emulsions were produced with Accosize 18 and Accosize 72 cationic starch (1:3 starch) as an emulsifier dissolved in water.

Air-formation

Figure 2 shows a schematic of the forming apparatus used to make the air-laid sheets (6). Sheets were either 100 percent cellulose fiber or 70/30 wt.% cellulose/polypropylene. Enough fiber for one 205 g/m^2 sheet (plus about 10% extra to compensate for "lost" fiber) was added to the top agitation chamber. A vacuum of 7 to 10 kPa was applied to the plenumat the bottom. Air jets in the agitation chamber were alternately pressurized by 500 kPa compressed air, forcing the fibers through an 8-mesh screen and down through the tower. The fibers collected on the forming screen at the bottom of the tower, creating a web of fibers. This web can be easily removed and pressed into a sheet.

For composite sheets containing polypropylene, static and clumping proved to be a problem at times in the formation process. To help break apart any polypropylene clumps prior to sheet air-formation, it was run through a Nit-Separator, designed at the USDA Forest Products Lab. (Fig. 3) (6). The "accepts" that passed through the 16-mesh separator screen were next mixed with water in a Waring blender to eliminate static surface charge, The fiber was dried before use.

Wet-laid handsheet formation

Prior to handsheet formation, the cellulose pulp was beaten (Valley) to approximately 500 Canadian Standard Freeness. For all sheets, the fibers were mixed in a bucket with water and allowed to soak overnight before sheet formation. Handsheets were formed by Tappi method T205 am-88 (20), but with tap water substituted for deionized water. Tap water has been found to increase the rate of AKD reaction (9),

Treatment methods

Different AKD and ASA treatments were applied to the air-formed sheets and wet-formed handsheets. Cellulose fiber used for the air-formed sheets was treated prior to sheet formation at a 1.5 wt.% (by cellulose fiber weight) level. This treatment level should be more than adequate to ensure complete reaction (10, 13). In fact, excess unreacted AKD has been shown (10) to contribute to the sizing process. Each treatment was prepared and poured over batches of 30 to 35 ovendry grams of fiber and stirred for 2 minutes. The excess liquid was drained off using a Buchner funnel and the resulting fiber was broken apart by hand into a large pan and dried in a 105°C oven for 1 hour.

In the case of handsheets, ring-dried handsheets were subjected to each treatment for 5 minutes (1.5 wt.% of cellulose fiber). Sheets were then allowed to air dry in a laboratory hood for 30 minutes, followed by 1 hour in a 105°C oven.

Sheet annealing

Some of the wet-formed handsheets and air-formed

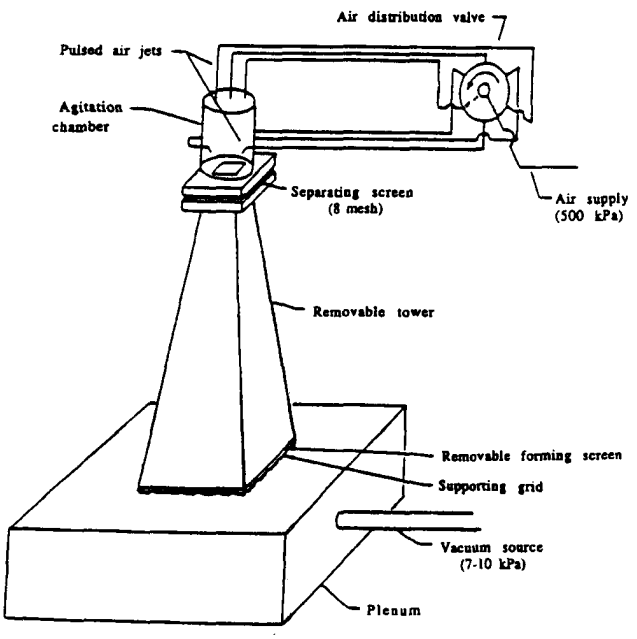


Figure 2. — Schematic diagram of air-forming apparatus.

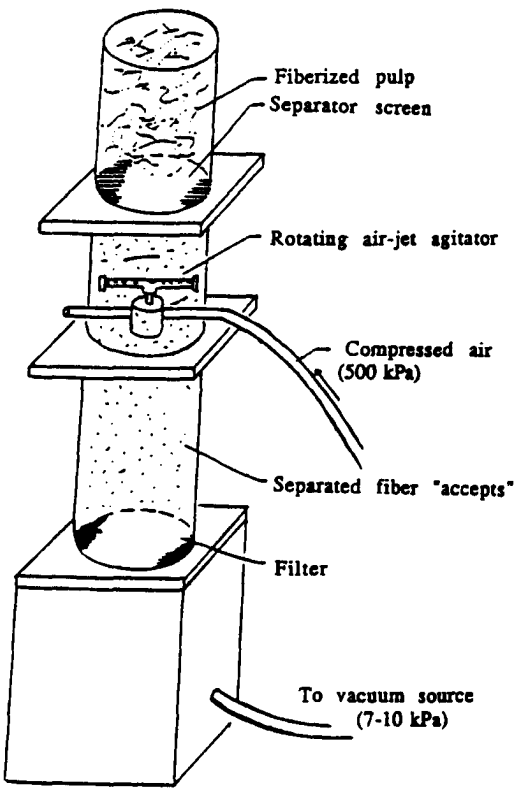


Figure 3. — Schematic diagram of FPL-designed Nit-Separator.

sheets produced and treated as above underwent an additional step. Prior to pressing, the sheets were placed in a circulating oven for 5 minutes at 130°C. Sheets were then pressed as described below.

Sheet pressing

Prior to pressing the 100 percent cellulose air-formed sheets, they were placed in a 90% RH room for at least 96 hours to increase the moisture content of the fibers in hopes of increasing the amount of hydrogen bonding in the sheets. No other sheets underwent this step.

All handsheets and air-formed webs were placed between two 9- by 9-inch stainless steel plates covered with a thin Teflon film and pressed in a Carver laboratory press at approximately 172°C and 350 psi (2260 Kpa) for 2 minutes. Upon removal from the press, the plates were sandwiched between a large steel block (bottom) and another (ca. 1 kg) steel plate (top) for 2 minutes to help prevent wrinkling in the sheets. After pressing, sheets were preconditioned in a controlled room (30 ± 5°C, 25 ± 10% RH) for at least 24 hours before being moved to a conditioning room (23 ± l°C, 50 ± 2% RH) where they remained for at least 24 hours

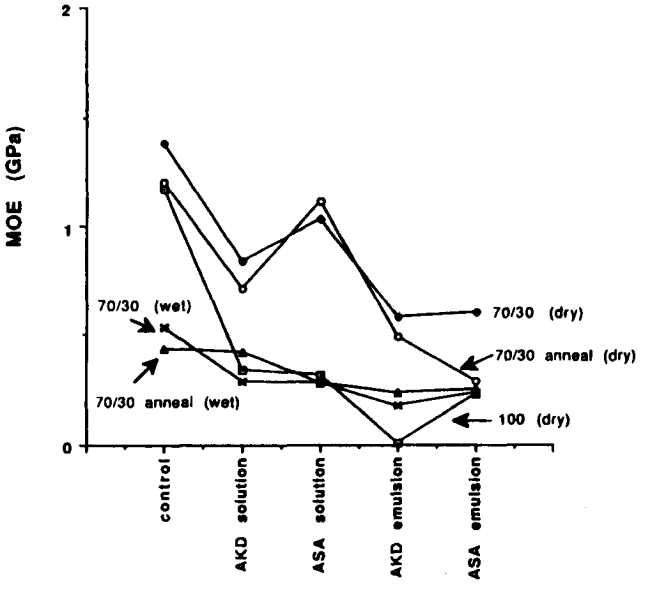


Figure 4. — Effect of treatments on modulus of elasticity for air-formed sheets.

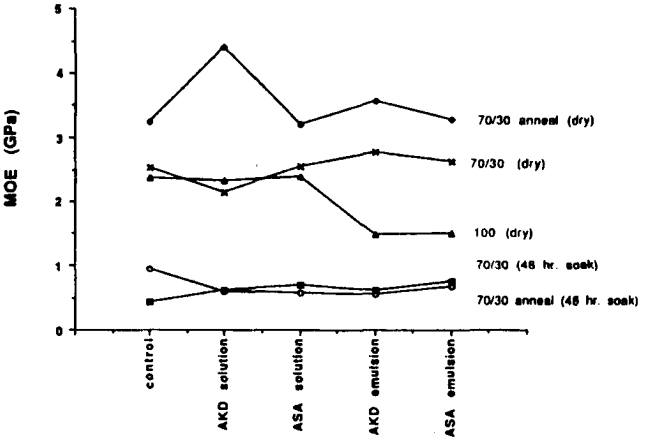


Figure 6. — Effect of treatments on modulus of elasticity for wet-laid handsheets.

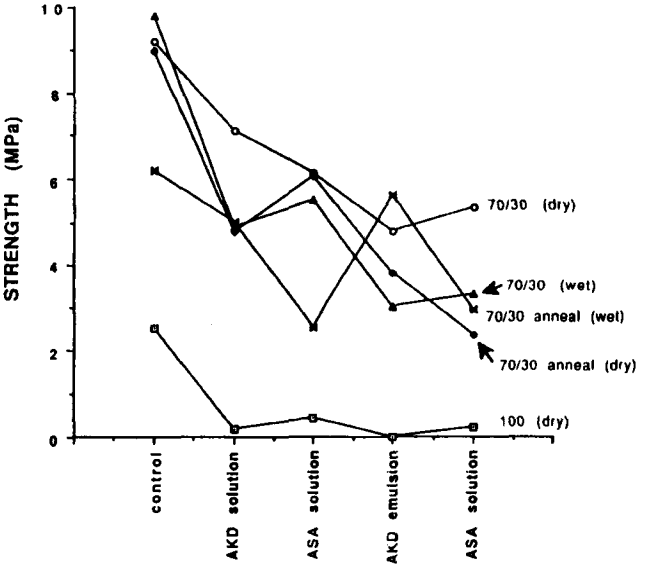


Figure 5. — Effect of treatments on breaking strength for air-formed sheets.

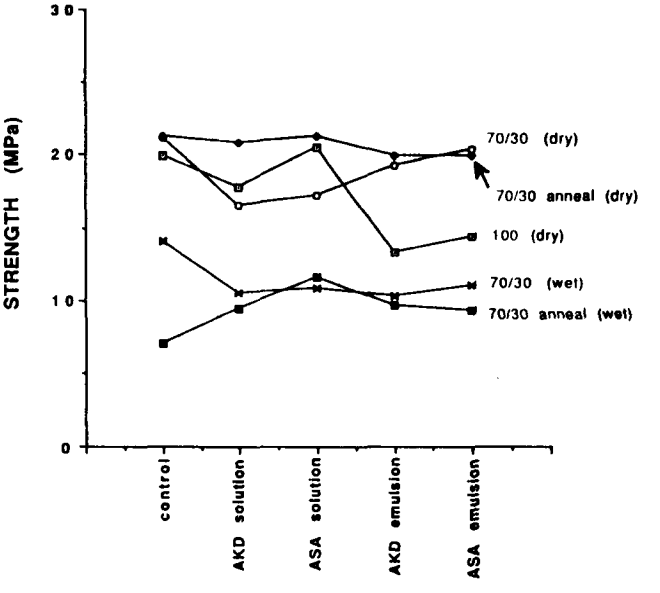


Figure 7. — Effect of treatments on bending strength for wet-laid handsheets.

before testing. Sheets used for 48 hour-soak samples did not undergo the preconditioning/conditioning step.

48-hour soak

To test the water durability of treated composite sheets, samples were submerged in approximately 1 inch of distilled water for 48 hours. Samples were then blotted and tested.

Mechanical and physical tests

Burst testing was done using a Mullen Tester Model CA (Perkins & Son, Inc.). Reported data are the averages of 8 to 10 samples.

A Taber V-5 Stiffness Tester was used to measure sheet stiffness, using 14 to 16 samples for each data point.

Internal bond measurements were done slightly differently than Tappi method T541 pm-83 (20). Instead of pressure-sensitive tape, heat-sensitive film (MT5 permanent Dry Mounting Tissue, Seal, Inc.) was used. Samples size was 1.75 by 1.75 inches (with test area of 0.785 in^2). Film bonding was done at 120°C for 45 minutes. Specimens were tested on an Instron Tester (Model TTCM) at 0.2 cm/min. crosshead speed. Data are the average of 27 to 30 samples.

Other mechanical properties (tensile modulus, tensile strength, elongation-at-break, tensile energy absorption) were measured using necked-down specimens and an Instron Tester (Model 4201). The crosshead speed was 3 mm/min. Properties were calculated by a regression program, unless otherwise stated, with reported data the average of 14 to 16 samples.

Density calculations were done by measuring the weight and average thickness of 3- by 3-inch or 4- by 4-inch samples. Thicknesses were measured using an effective thickness micrometer, described elsewhere (18). Values reported are the average of five measurements.

Results

Figures 4 through 7 summarize results obtained from tensile test measurements and Tables 1 and 2 give other testing results.

Table 1. — *Summary of data for air-formed sheets Number in parentheses are standard deviations.*

	Stiffness	Burst strength	Internal bond	Density	Strain at break (E_{max})	Tensile energy absorption
	(g - cm)	(psi)		(g/cm^3)	(%)	(MPa)
100 (dry)						
Control	4.5 (0.9)	3.4 (0.8)	21.1 (15.4)	0.72	0.34 (0.09)	.0051 (.0024)
AKD solution	0.4 (0.3)	- -[a]	0.7 (0.2)	0.48	1.17 (0.49)	.0015 (.0009)[c]
ASA solution	1.0 (0.6)	- -[a]	0.9 (0.6)	0.60	1.02 (0.52)	.0027 (.0012)
AKD emulsion	0.3 (0.1)	- -[a]	0.6 (0.1)	0.57	- -[a]	- -[a]
ASA emulsion	0.6 (0.4)	- -[a]	3.8 (3.7)	0.62	1.08 (0.61)	.0017 (.0012)[c]
70/30 (dry)						
Control	7.6 (1.7)	23.0 (7.0)	161.7 (61.5)	0.66	4.88 (2.05)	0.40 (0.23)
AKD solution	5.5 (1.5)	24.1 (4.9)	121.5 (43.1)	0.66	4.52 (1.57)	0.27 (0.16)
ASA solution	7.1 (1.9)	32.3 (8.6)	89.9 (26.7)	0.66	4.05 (1.03)	0.20 (0.08)
AKD emulsion	3.3 (1.1)	11.3 (2.6)	6.1 (2.7)	0.53	3.41 (0.88)	0.13 (0.05)
ASA emulsion	3.7 (1.0)	13.9 (5.3)	10.9 (5.7)	0.61	3.13 (1.02)	0.12 (0.06)
70/30 (48 hr. soak)						
Control	2.9 (0.7)	39.5 (9.2)			9.69 (0.42)	0.65 (0.12)
AKD solution	3.4 (1.2)	20.6 (7.4)			8.53 (1.65)	0.30 (0.07)
ASA solution	3.1 (0.8)	27.5 (13.1)			9.26 (0.90)	0.36 (0.10)
AKD emulsion	2.0 (0.6)	13.5 (4.6)			5.67 (2.04)	0.13 (0.08)
ASA emulsion	1.6 (0.6)	13.1 (4.6)			4.64 (1.74)	0.11 (0.05)
70/30 anneal (dry)						
Control	13.9 (2.5)	33.7 (6.7)	81.0 (31.6)	0.61	5.45 (1.13)	0.41 (0.12)
AKD solution	12.0 (2.5)	18.3 (2.9)	24.5 (12.5)	0.51	4.39 (1.45)	0.17 (0.06)
ASA solution	16.4 (3.8)	23.7 (5.9)	64.3 (30.9)	0.58	3.39 (1.42)	0.16 (0.08)
AKD emulsion	3.2 (1.2)	9.2 (1.7)	9.0 (4.5)	0.55	2.88 (0.62)	0.08 (0.03)
ASA emulsion	3.4 (1.3)	6.4 (2.3)	9.6 (3.3)	0.51	2.23 (0.60)	0.04 (0.02)
70/30 anneal (dry)						
Control	2.4 (0.4)	30.9 (9.5)			8.51[b]	0.37 (0.09)
AKD solution	3.3 (0.8)	22.0 (6.7)			7.94[b]	0.29 (0.08)
ASA solution	3.9 (1.0)	28.5 (9.0)			3.23 (1.02)	0.06 (0.03)
AKD emulsion	1.6 (0.5)	7.8 (3.1)			7.68[b]	0.31 (0.10)
ASA emulsion	1.7 (0.6)	7.4 (2.1)			4.51 (1.97)	0.10 (0.06)

[a] Too low to measure.
[b] Maxed out strain gauge on several samples. Actual value is probably higher than reported.
[c] Estimated from tensile curves.

Dry properties

As can be noticed, properties of air-formed sheets are in all cases significantly less than those of the handsheets. Also, AKD and ASA treatments nearly always had a detrimental or only slight positive effect on properties if the sheets had not been annealed. Annealing the wet-formed handsheets increased modulus, strain-to-break, and burst strength, but had little positive effect on other dry sheet properties. For air-formed sheets, annealing seems to have a minimal to negative effect on sheet properties.

For 70/30 composite air-formed sheet that were not annealed before pressing, the relatively low density (compared to handsheets) was little affected by the change in fiber surface character, but sheets made that included the annealing step show a decrease in density if treated. Wet-formed composite handsheets showed a 4 to 11 percent decrease if they were not annealed before pressing, while a slight increase was seen for annealed sheets.

Water-soaked properties

The wet strength tensile properties (modulus and strength) of unannealed treated composite sheets, either wet-laid or air-formed, generally showed a greater percentage of decline than did those for untreated control sheets, while other sheet properties tended to show equal declines. For wet-laid handsheets, this trend is reversed if the sheets underwent the annealing step before pressing. Here mechanical properties of treated sheets retain a greater percentage of their original value than do untreated sheets.

For the air-formed sheets, annealing seems to be slightly beneficial, but properties are not dramatically different than annealed control values.

Because of specimen preparation methods, internal bond testing can not be performed on water-soaked samples.

Emulsion versus solution

For the air-formed sheets, either of the emulsion treatments gave much poorer sheet properties than did the solution treatments. In the case of wet-formed handsheets, the difference between emulsion and solution treatments appears to be minimal, although solution treatment may give slightly better properties in 70/30 annealed sheets, while emulsion treatment being better in 70/30 unannealed sheets.

Internal bond tests were not done on water-soaked sheets.

Table 2. — *Summary of data for wet-laid handsheets Number in parentheses are standard deviations.*

	Stiffness	Burst strength	Internal bond	Density	Strain at break (E_{max})	Tensile energy absorption
	(g - cm)	(psi)		(g/cm^3)	(%)	(MPa)
100 (dry)						
Control	24.6 (1.0)	49.1 (2.8)	115.4 (14.6)	0.68	3.82 (0.76)	0.59 (0.13)
AKD solution	28.9 (2.0)	50.8 (2.5)	105.6 (5.8)	0.63	3.74 (0.50)	0.53 (0.09)
ASA solution	29.5 (3.7)	69.2 (6.2)	99.0 (6.9)	0.64	4.20 (0.43)	0.67 (0.09)
AKD emulsion	19.9 (3.3)	48.9 (6.6)	46.3 (7.0)	0.60	4.35 (0.73)	0.45 (0.10)
ASA emulsion	22.2 (5.4)	57.3 (10.2)	57.4 (3.8)	0.63	4.46 (0.66)	0.50 (0.10)
70/30 (dry)						
Control	17.7 (1.7)	51.5 (16.3)	217.2 (23.7)	0.81	2.66 (0.59)	0.42 (0.14)
AKD solution	21.7 (2.0)	43.3 (7.0)	182.4 (30.2)	0.72	2.68 (0.78)	0.34 (0.14)
ASA solution	26.4 (4.8)	52.9 (11.5)	187.7 (21.3)	0.73	2.53 (0.92)	0.34 (0.15)
AKD emulsion	23.8 (2.5)	63.3 (9.9)	154.8 (24.5)	0.77	3.68 (0.92)	0.56 (0.17)
ASA emulsion	23.0 (3.4)	74.6 (13.3)	169.9 (24.1)	0.78	4.58 (1.07)	0.74 (0.22)
70/30 (48 hr. soak)						
Control	4.1 (1.0)	48.1 (9.2)			7.40 (0.82)	0.66 (0.14)
AKD solution	4.5 (0.7)	42.2 (5.6)			8.28 (1.39)	0.55 (0.20)
ASA solution	4.2 (0.8)	53.6 (12.1)			8.00 (0.85)	0.54 (0.11)
AKD emulsion	4.5 (0.5)	48.5 (10.7)			8.71 (0.85)	0.55 (0.12)
ASA emulsion	3.7 (0.6)	42.7 (6.0)			8.30 (1.08)	0.56 (0.11)
70/30 anneal (dry)						
Control	21.9 (3.4)	63.8 (11.1)	190.1 (26.8)	0.69	4.38 (0.65)	0.74 (0.16)
AKD solution	27.2 (3.7)	68.9 (10.2)	196.4 (21.8)	0.70	3.47 (0.76)	0.58 (0.18)
ASA solution	26.5 (4.5)	76.4 (7.9)	181.7 (27.1)	0.73	4.56 (0.76)	0.76 (0.18)
AKD emulsion	23.2 (2.5)	68.4 (12.8)	156.7 (29.9)	0.71	4.78 (0.56)	0.76 (0.12)
ASA emulsion	23.3 (2.8)	82.6 (11.1)	167.5 (14.9)	0.72	4.72 (0.83)	0.75 (0.19)
70/30 anneal (dry)						
Control	3.9 (0.5)	36.4 (7.6)			6.90 (0.87)	0.30 (0.06)
AKD solution	4.4 (0.8)	38.6 (6.9)			7.34 (1.01)	0.43 (0.09)
ASA solution	4.4 (0.7)	49.5 (11.1)			8.14 (0.68)	0.58 (0.08)
AKD emulsion	4.1 (0.9)	47.5 (7.5)			7.86 (0.86)	0.45 (0.10)
ASA emulsion	3.9 (0.8)	42.3 (8.9)			7.80 (1.00)	0.45 (0.10)

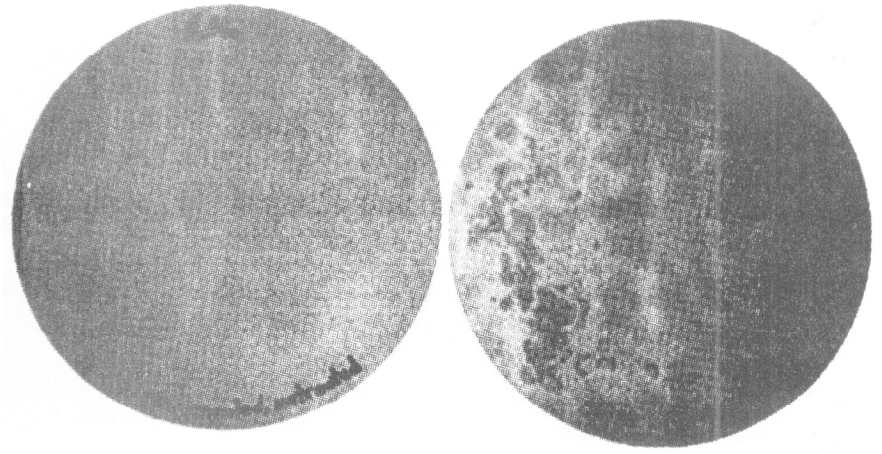


Figure 8. — Photo of formation differences between untreated annealed and untreated unannealed 70/30 handsheets.

Discussion

It is apparent that the effects of AKD and ASA treatments on the adhesion of polypropylene to cellulose are generally not positive. As the sheet densities seem to show, the treatments appear to prevent or decrease intimate contact between the polypropylene-cellulose or cellulose-cellulose fibers unless the wet-laid sheets were annealed before pressing.

As might be expected, the air-formed, 100 percent cellulose sheet, held together mainly by weak hydrogen bonding between "dry" fiber, felt the largest effect of the treatments, which covered the hydrophilic cellulose surface with hydrophobic molecules prior to web formation. This was less destructive in the wet-formed 100 percent cellulose handsheets, which had hydrogen bonding established prior to treatments.

Visual inspection of the pressed handsheets revealed a rather startling result. Unannealed untreated pressed composite handsheets appeared to have polypropylene-rich segregated areas. But in handsheets that underwent a treatment or annealing before pressing, these areas seemed to be eliminated, as can be seen in Figure 8. This suggests that the treatments (or annealing) had the effect of compatibilizing the surface energies of the two components, facilitating spreading of the molten polypropylene. This appears to be offset by the loss in hydrogen bonding (which provides a significant portion of the strength) between cellulose fibers with hydrophobic surfaces, and yields the overall decline in mechanical properties.

A probable explanation for the poor properties seen in the emulsion-treated air-formed sheets is the inhomogeneity of the pressed sheets. After the cellulose fiber is treated, drying in the oven results in the formation of fiber clumps. While passing through the air-forming apparatus, many of these clumps do not break apart, resulting in poor sheet quality.

Effects of annealing

It was found during a simple water-submersion test of the polypropylene that heating the polypropylene at 130°C for 5 minutes noticeably increased the submersion time, while heating at 105°C for up to 1 hour did not. ESCA analysis of the polypropylene fibers before and after the 130°C heating revealed an increase by nearly a factor of three in the amount of surface oxygen after heating. This corresponds with thermal oxidation of the polypropylene, which has been shown (1,16) to produce various products on the polymer, such as ketones, esters, and acids, This increase in the polar nature of the polypropylene surface is possibly responsible for the increased properties of the annealed handsheets.

The fairly small hydrocarbon chain length (R=C12-C18) of the AKD and ASA treatments, although efficient at changing the hydrophilic nature of the cellulose surface, may not be long enough to be truly incorporated into the polypropylene, resulting in poor adhesion between the two components. Other studies involving maleated polypropylene (5,14,19), a compound strikingly similar to ASA but with a longer chain, have shown it to be an effective coupling agent for wood-polypropylene composites.

Conclusions

Air-forming composite sheets instead of traditional wet handsheet formation does not provide any additional benefits to the mechanical properties of the sheets, and in fact yields poorer quality sheets. AKD and ASA, in either emulsion or solution form, have little beneficial effect on the mechanical properties of both dry-tested air-formed sheets and dry-tested wet-formed handsheets. Annealing the wet-laid handsheets prior to pressing increases properties over unannealed handsheets, but treatments still have little effect on dry-tested properties. For air-formed sheets, annealed and unannealed sheets are nearly identical in both wet and dry tests. While sheet treatments and annealing do appear to influence the spreading of polypropylene on cellulose, the increased hydrophobicityof the cellulose surface (10,13) appears to destroy important hydrogen bonding in the sheets, leading to a decrease in properties, In addition, water-soaked samples showed no additional moisture resistance because of AKD or ASA treatment, even though the cellulose fiber surfaces had become more hydrophobic.

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