〔목재공업 11(3) : 3-13(1983) (論文) 〕

The Application of Rule of Mixtures to Fiber-Reinforced Composites(1)[*1]

– Mechanical Properties of Fiber-Reinforced, Sulfur-Based Composites –

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목재 섬유 複合材에 混合理論의 적용에 關한 研究 (1)[*1]

– 硫黃 化合物을 사용한 木材 纖維 複合材의 기계적 性質 –

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要 約

크라프트 펄프의 screening rejects, 볏짚 그리고 이들의 1:1 비율의 혼합 물질을 사용하여, 5 가지의 다른 纖維板 密度를 가지는 섬유판을 만들었다. 이들을 硫黃 化合物에 沈積시켰다. 製造한 複合材 속의 硫黃 化合物의 老化効果를 관찰하기 위해 1 년동안 일정한 時間 間隔으로, 이 복합재의 기계적 強度를 Young 係數로 나타내었다.

最適의 섬유판과 유황 화합물의 제조 조건下에서, 이 木材 纖維 複合材의 Young 係數는 기존의 합성수지로 만든 복합재나 목재 섬유로 만든 集成材나 복합재보다 훨씬 큰 결과를 보여 주었다. 예를 들어, 목재 섬유판 밀도가 0.35gm/cm^{3}인 이 복합재의 modulus of elasticity와 modulus of rupture는 각각 1,000,000psi 와 7000psi인데 반해, 섬유판 밀도가 1.28gm/cm^{3}인 hardboard의 그것들은 각각 800,000psi 와 6000psi를 나타내었다.

Summary

Fiber mats were made at five density levels, using fibers from kraft pulp screening rejects, rice straw and a 50/50 mixture of the two. They were soaked in the sulfur compounds. Specimens cut from the composite panels were tested in flexure at time intervals for one year to study the effect of aging. Modulus of elasticity (MOE) and modulus of rupture (MOR) were determined.

Under optimum conditions of fiber mat preparation and saturation with molten sulfur and modified sulfur, composites were produced which exhibited mechanical properties comparable to conventional fiberglass in some properties and superior to conventional wood-based composition boards. For example, the moduli of elasticity of the reinforced composites made from pulp screening rejects, with a density of 0.35 gm/cm^3, were greater than 1,000,000 psi, as compared 800,000 psi for high-density hardboard (1.28 gm/cm^3). Modulus of rupture of the best reinforced composites was about 7,000 psi, comparable to 6,000 psi of high-density hardboard.

1. Introduction

Composite materials have a long history of usage. Their beginnings are unknown, but all recorded history contains references to some form of composite material. For example, straw was used by the Israelites to strengthen mud bricks. Plywood was used by the ancient Egyptians when they

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realized that wood could be rearranged to achieve superior strength and resistance to dimensional change caused by swelling or shrinkage due to moisture change. So the advantage of composites is that they usually exhibit the best qualities of their constituents and often some qualities that neither constituent possesses. The properties that can be improved by forming a composite material include: strength, stiffness, corrosion resistance, fatigue life, thermal insulation and thermal conductivity.

The composite made by impregnation of liquid sulfur into wood or wood fiber is not new. Ellis (1912) used high-sulfur asphalt to introduce sulfur into wood. He also used hydrocarbons which can be saturated with up to 10 percent sulfur at 100°C to impregnate wood. Upon cooling, the solubility decreases, and the excess sulfur precipitates in situ. Kobbe (1926) immersed wood for several in hours liquid sulfur at 140°−150°C until all water had boiled off.

Figure 1 shows the viscosity-temperature curve for liquid sulfur (Fanelli and Bacon, 1943).

The liquid sulfur below 159°C consists primarily of S_8 rings concerning which at least 19 different melting points of sulfur have been published. Obviously, the equilibrium composition of the sulfur melt is not yet established. The natural thermodynamic melting point is difficult to determine because of slow kinetics. It is now assumed to be 119.6°C (Thackray, 1965).

When molten sulfur is heated to a temperature around 159°C, there is quite an abrupt and very large increase in viscosity, followed by a gradual decrease at higher temperatures, as shown in Figure 1. The temperature of 159°C is called the transition temperature.

The three classes of composite materials are: dispersion-strengthened composite materials, particle-reinforced composite materials and fiber-reinforced composite materials. They are distinguishable by their microstructures. It is known that dispersion-strengthened composite materials are characterized by a microstructure consisting of an elemental or alloy matrix within which fine particles of 0.01 to 0.1 μ in diameter are uniformly dispersed in a volume concentration of 1 to 15%. Particle-reinforced composites differ from the dispersion-strengthened composites since the dispersoid size exceeds 1.0 μ and the dispersoid concentration exceeds about 25%. The distinguishing microstructural feature of fiber-reinforced materials is that their reinforcement has one long dimension, whereas the reinforcement particles of the other two composites do not. The reinforcing phase in fiber composite materials spans the entire range of size, from a fraction of a micron to several mils in diameter, and the entire range of volume concentrations, from a few percent to greater than 70 percent (Broutman and Krock, 1967).

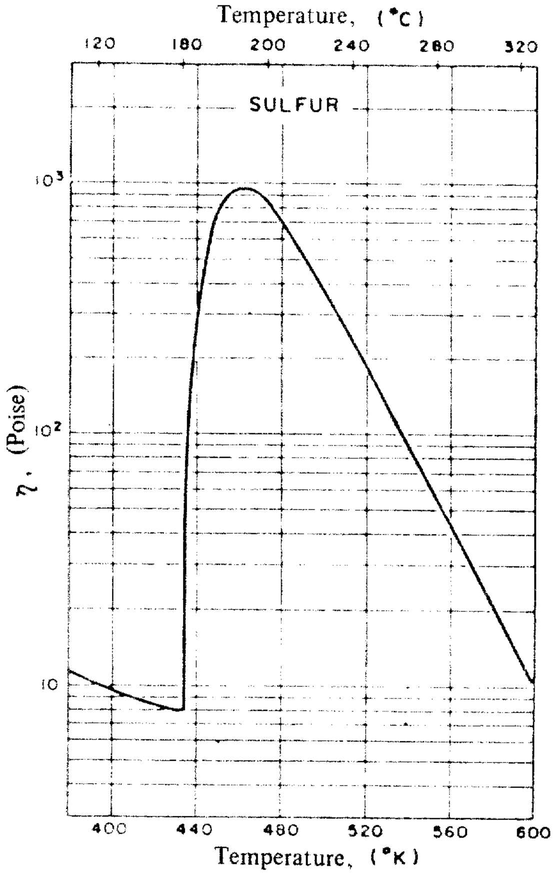

Fig. 1. Viscosity-temperature curve for liquid sulfur (Fanelli and Bacon, 1943).

Modification of sulfur with organic materials has a wider practical application than with inorganic material. There are many polyfunctional aromatic and aliphatic materials available which are easily reacted with elemental sulfur. Chlorobenzene, limonene, myrcene, alloocine, dicyclopentadiene, cyclododeca-1, 5, 9-triene, cycloocta-1, 3-diene, styrene and the polymeric polysulfides, Thiokol LP-31, -32, and -33 are well known organic materials that react with sulfur (Macallum, 1951, Lentz and Carrington, 1959).

Each of the above modifiers reacts with excess sulfur at 140°C to give a mixture of polysulfides and unreacted sulfur. Even though in some cases substantial amounts of this unreacted sulfur may be held indefinitely in a metastable condition as monoclinic sulfur, or "S_8 liquid", the percentage of this unreacted material increases with storage time. The reaction mechanisms of several representative modifiers with sulfur are shown below.

(i) Dichlorobenzene

Figure 2 shows elemental sulfur and dichlorobenzene react with each other with elimination of hydrogen chloride, whereby polymeric phenylene polysulfides are formed. The average number of sulfur atoms between the phenylene groups depends on the amount of sulfur in the reaction mixture and may vary from 2 to 7 (Lentz and Carrington, 1959).

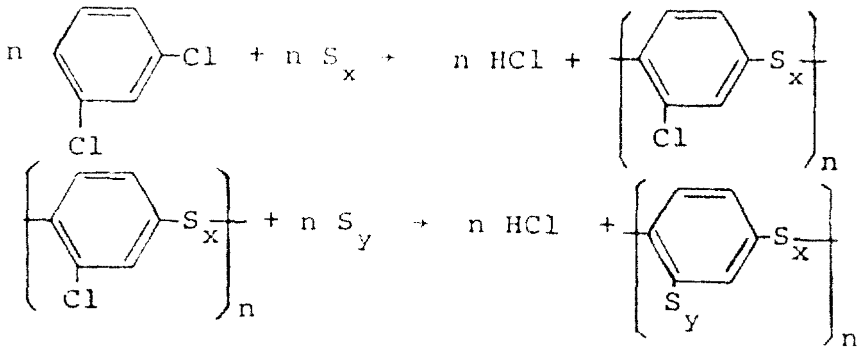

Fig. 2. The reaction mechanism between elemental sulfur and dichlorobenzene (Lentz and Carrington, 1959).

(ii) Dicyclopentadiene

The beneficial use of dicyclopentadiene to modify sulfur has been reported by a number of workers including Currell et al. (1975), Sullivan et al. (1975), and also Diehl (1976). Figure 3 shows that the interaction of dicyclopentadiene and elemental sulfur at 140°C gives a mixture of polysulfides and free elemental sulfur.

Currell et al. (1978) summarized the insoluble fraction of sulfur modified by dicyclopentadiene in carbon disulfide solvent at 140°C (Table I). They indicated that in every case all the unreacted

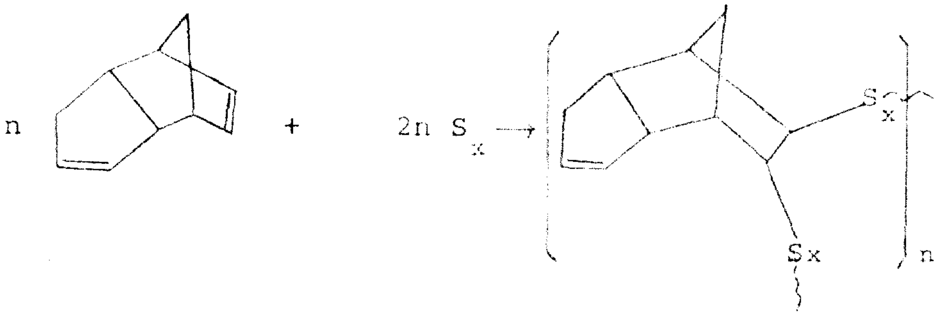

Fig. 3. The reaction mechanism between elemental sulfur and dicyclopentadiene (Blight et al., 1978).

free sulfur was soluble in carbon disulfide, indicating that it is non-polymeric and presumably has a ring structure. The amount of insoluble material formed increases with reaction time. This insoluble fraction may be a high-molecular-weight, cross-linked material, which swells in carbon disulfide.

(iii) Styrene

As with the dicyclopentadiene reaction, Figure 4 shows the reaction product with styrene is also a mixture of unreacted free sulfur and polysulfides. The reaction of sulfur with styrene is exothermic, a temperature rise being observed for about 30 minutes. However, in contrast to the dicyclopentadiene, the reaction mixture may be stirred easily thereafter (Blight et al., 1978). When this modified sulfur is compared with that modified by dicyclopentadiene, it is assumed that the proportion of polysulfides to styrene is much lower than in the dicyclopentadiene modified sulfur at the same level of modifier loading and heating conditions. In other words, a more crystallized and brittle product is obtained when sulfur is modified with styrene.

Table 1. Insoluble fraction of sulfur modified by dicyclopentadiene* in carbon disulfide solvent

(Currell et al., 1978)

Modifier Loading (%)	Heating Time (hours)	Fraction Insoluble in CS_2 (%)
5	3	11.4
10	3	13.4
25	3	15.1
25	20	56.6

*After storage for 18 months at ambient temperature. Percentage figures given each refer to percentage of total composition.

2. Experimental procedure

A series of fiber mats were prepared from hardwood kraft pulpmill screening rejects provided by the Crown Zellerbach pulpmill at Wallula, Oregon, rice straw pulp made from rice straw provided by a California rice grower and a 50/50 blend of each fiber type.

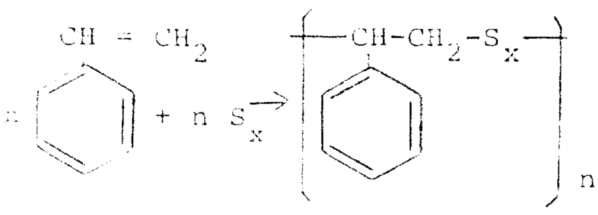

Fig. 4. The reaction mechanism between elemental sulfur and styrene (Blight at al., 1978).

The coarse kraft pulp rejects were passed through a laboratory model, 13-inch Sprout Waldron refiner to obtain a uniform fiber bundle size suitable for mat making. After refining the pulp was thoroughly washed.

Rice straw pulp was made by soaking chopped straw in a 1% NaOH solution overnight and beating it for about 2 minutes in a laboratory model Hollander. After beating, the pulp was thoroughly washed to remove pith and fines.

Fiber mats were made in a 12-inch-square sheet mold, using enough fiber to produce pressed mats of about 1/8-inch thickness at the required density. In order to control density, mats were first air dried and then pressed between screens in a steam-heated laboratory hot press using 1/8-inch-thick metal stops at the edges of the mats.

Pressed fiber mats were cut into 2-inch by 5-1/2-inch specimens. These were immersed in molten sulfur, or in molten modified sulfur, at about 135°-140°C until they were thoroughly saturated. The time required for thorough saturation varied from 10 to 30 minutes.

3. Results and discussion

There are several factors which may influence the MOE and MOR of composites made from different types of fiber.

For example, even though the submicroscopic anatomy of rice straw apparently has not been reported, it is well known that the angle of microfibrils in the secondary wall in wood fiber influences both the mechanical and physical properties (Panshin and de Zeeuw, 1964). It follows that where microfibrils of cellulose are nearly parallel to the fiber axis the modulus of elasticity and the tensile strength are higher than where the fibril angle is great. Thus the higher mechanical strengths of composites made with pulp screening rejects may be due to a difference in the fibril angle between wood fiber and rice straw fiber.

Table 2. Comparison of some properties of typical fiber-reinforced, sulfur-based (FRSB) composites with other composites and with wood-based composition boards (Bryant and Lee, 1981)

Materials	Specific gravity (%fibre)	MOE, psi (M pascals)	MOR, Psi (K pascals)
FRSB composites with unmodified sulfur			
Kraft pulp rejects	4.40	1,400,000	7,350
	(25.%)	(9,000	(50,000)
Kraft pulp rejects	0.25	650,000	4,080
	(18%)	(4,500)	(28,200)
Rice straw	0.40	710,000	4,110
	(25%)	(4,900)	(28,500)
Coir (coconut husk)	0.20	329,000	8,800
	(13.3%)	(2,300)	(60,900)
Bagasse	0.20	674,000	5,000
	(17.5%)	(4,700)	(34,600)
Glass fibre/polyster			
Thermoplastic	1.52	1,150,000	26,000
	(30%)	*8,000)	(180,000)
Thermoplastic	1.48-1.67	800,000	26,000
	(10%)	(5,500)	(180,000)
Thermoset, preformed	1.35-2.30	1,000-300,000	10,-40,000
	(varies)	(700-2,100)	(69,277,000)
Wood-based composition boards			
Medium-density	0.80	400,000	3,000
		(2,000)	(20,800)
High-density hardboard	1.28	800,000	7,000
		(5,500)	(48,500)
Medium density particleboard	0.59	250,000	1,600
		(1,700)	(11,100)
High density particleboard	0.80	700,000	8,000
		(4,800)	(55,400)

Another important feature of the microstructure of cellulosic fibers which influence their mechanical properties is the thickness of the S_2 layer in the cell wall. This is the layer that contains the highly oriented cellulose, and the density of this cellulose reflects the relative proportion of cellulose to lignin, hemicellulose and extractives (Panshin and de Zeeuw, 1964). So it may be speculated that the higher MOE and MOR of composites made with pulp screening rejects over those made with rice straw are due to the higher cellulose content of the former.

Table II shows the typical mechanical properties of fiber-reinforced, sulfur-based composites compared with those of wood-based composition boards.

The wide range of values for the mechanical property results shown in Table III and IV reflects the influence of fiber type, fiber mat density and impregnant type after 60 and 360 days aging of the composites respectively. Table V and VI indicate that these three main effects were statistically significant for both modulus of elasticity and modulus of rupture for the composites of 60 days aging case. More interesting, however, were the significant two-way interactions involving combinations of two of the three variables.

Table 3. The modulus of elasticity** and modulus of rupture*** of impregnated specimens after 60 days aging

Type of impregnant / Type of fiber / Fiber density (gm/cm^3)	Pure sulfur			Pure crystex			Sulfur DCP**** (20%)			Sulfur + DCP (5%)			Sulfur STY***** (2%)			Sulfur + STY (5%)			Crystex + DCP (2%)		
	Pulp reject	*P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw
0.20	44.5	42.3	39.1	54.0	51.1	44.9	49.5	44.0	35.5	34.1	32.0	29.9	63.5	55.5	45.0	54.1	45.9	40.0	48.5	40.4	34.6
0.25	65.4	61.2	43.9	68.0	65.4	49.6	61.0	55.9	40.1	52.0	39.8	35.1	75.1	68.8	50.6	62.0	54.1	43.9	61.5	53.6	37.4
0.30	88.7	80.7	56.1	85.1	83.4	61.0	78.9	74.6	44.0	59.9	50.2	41.8	84.9	77.6	60.9	74.9	70.0	58.1	74.4	67.7	45.6
0.35	111	93.6	64.1	108.9	107.2	71.4	86.1	79.2	54.3	76.8	61.0	50.2	87.4	81.6	72.0	84.2	73.7	62.1	83.6	81.4	56
0.40	140	110	72.7	141	124	76.1	101.5	91.8	69.7	86.2	69.9	61.0	102.1	86.6	75.4	95.8	85.3	73.9	101.0	88.0	67.5

4. Conclusions

In order to investigate the mechanical and physical properties of fiber-reinforced, sulfur-based composites, some factors affecting the properties of the composites were considered. These were: fiber type, fiber mat density, the type and concentration of modifiers and the aging effect of the impregnants in the composites.

The effect of fiber types on mechanical properties (i.e. modulus of elasticity, MOE, and modulus of rupture, MOR) was significant. The MOE of the composites made from pulp screening rejects, with

Table 4.

Type of impregnant / Type of fiber / Fiber density (gm/cm^3)	Pure sulfur			Pure crystex			Sulfur + DCP (2%)		
	Pulp reject	P(50%) / S(50%)	Rice straw	Pulp reject	P(50%) / S(50%)	Rice straw	Pulp reject	P(50%) / S(50%)	Rice straw
0.20	28.0	22.1	18.9	29.1	28.4	21.7	37.8	33.0	21.2
0.25	37.1	28.9	23.0	39.0	34.5	27.0	41.7	39.0	29.7
0.30	54.0	44.2	39.1	55.1	49.5	39.6	53.2	42.6	32.0
0.35	58.8	52.0	46.2	60.9	54.8	49.4	53.0	50.8	38.8
0.40	61.1	57.2	47.8	66.2	59.7	54.2	38.5	57.8	44.5

Fiber density (gm/cm^3)	Sulfur + DCP (5%)			Sulfur + STY (2%)			Sulfur + STY (5%)			Crystex + DCP (2%)		
	Pulp reject	P(50%) / S(50%)	Rice straw	Pulp reject	P(50%) / S(50%)	Rice straw	Pulp reject	P(50%) / S(50%)	Rice straw	Pulp reject	P(50%) / S(50%)	Rice straw
0.20	32.1	24.8	18.1	38.3	34.2	21.8	41.0	36.0	22.3	38.2	34.3	22.3
0.25	34.9	29.6	20.5	43.1	39.0	30.8	44.5	39.5	30.7	41.5	39.5	31.2
0.30	46.0	40.4	22.6	50.7	43.3	32.5	51.2	45.3	34.8	54.0	43.2	33.0
0.35	48.1	42.8	33.1	56.8	52.0	39.0	58.2	52.7	40.8	54.8	51.2	39.5
0.40	51.8	50.2	33.9	61.5	55.8	44.7	62.0	53.8	45.2	58.7	58.8	44.5

* P = pulp reject, S = rice straw, ** MOE (psi x 10^4), *** MOR (psi x 10^2)

**** DCP = dicyclopentadiene, ***** STY = styrene

Table 4. The modlulus of elasticity and modulus of rupture of impregnated specimens after 12 months aging

Type of impregnant / Type of fiber / Fiber density (gm/cm^3)	Pure sulfur			Sulfur + DCP (5%)		
	Pulp reject	P(50%) / S(50%)	Rice straw	Pulp reject	P(50%) / S(50%)	Rice straw
0.20	47	42	39	38	34	30
0.25	66	61	45	55	44	40
0.30	90	81	56	69	57	48
0.35	111	94	65	88	67	56
0.40	142	110	75	94	76	66

a density of 0.40 gm/cm^3, was greater than 1,300,000 psi, as compared with 700,000 psi for the composites made from rice straw with the same density. At the same time the MOR of the composites made from pulp screening rejects with the same density was about 6000 psi, as compared to about 5000 psi for the composite made from rice straw.

The effect of fiber mat density upon MOE and

MOR was also significant. Generally the MOE of composites made with a fiber mat density of 0.40 gm/cm^3 were almost 2.5 times greater than those with a density of 0.20 gm/cm^3. On the other hand, the composites with the highest mat density (0.40 gm/cm^3) resulted in MOR values almost twice as high as those made with a mat density of 0.20 gm/cm^3 whatever impregnant was used.

The mechanical properties of fiber-reinforced, sulfur-based composite were affected by the crystallization of impregnated pure and modified sulfur in the composites. This crystallization changed with aging time after which the mechanical properties stabilized. Generally both MOE and MOR increased as the crystalline sulfur component increased with aging time. Accordingly the time required for stabilization depended on the concentration and types of modifiers used to modify the sulfur impregnant. Flexure test results showed that pure

Sulfur + DCP (20%)			Sulfur + STY (5%)			Sulfur + STY (20%)		
Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw
33	30	25	56	48	44	56	48	42
48	41	36	66	58	47	65	60	55
62	54	48	78	72	58	76	74	71
80	69	55	90	80	68	90	82	77
88	72	64	105	92	77	110	100	87

Type of impregnant / Type of fiber / Fiber density (gm/cm^3)	Pure sulfur			Sulfur + DCP (5%)		
	Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw
0.20	29	25	19	37	26	22
0.25	39	32	26	37	33	28
0.30	54	46	40	47	43	30
0.35	60	53	48	50	45	34
0.40	66	58	52	54	52	37

Sulfur + DCP (20%)			Sulfur + STY (5%)			Sulfur + STY (20%)		
Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw	Pulp reject	P(50%)/S(50%)	Rice straw
25	21	18	42	38	25	42	37	26
30	24	20	45	42	31	46	42	32
35	29	25	54	44	37	54	44	42
38	34	27	60	53	44	60	53	45
44	37	31	65	56	47	67	56	50

Table 5. Master table analysis of variance MOE of specimens tested after 60 days aging

Source of variation		Gross SS-CT	Net SS	df	Mean sq. variance	F ratio	Sig.
Total	G	528261					
	-CT	474257	54,004	104			
Treat-	G	481590	7,333	6	1222	42.4	***
ment	-CT	474257					
Density	G	503676	29,419	4	7355	255.4	***
	-CT	474257					
Fiber	G	485759	11,502	2	5751	199.7	***
Type	-CT	474257					
Tr.x Den.	G	513015					
	-CT	474257	2006	24	84	2.9	***
-Net SS	Tr.	7333					
-Net SS	Den.	29419					
Ft.x Den.	G	516634					
	-CT	474257	1456	8	182	6.3	***
-Net SS	FT	11502					
-Net SS	Den.	29419					
Tr.x Ft.	G	493998					
	-CT	474257	906	12	76	2.6	***
-Net SS	Tr.	7333					
-Net SS	Ft.	11502					
Error by subtraction			1382	48	28.8		

Includes: Tr.x Den.x Ft.

Separate F test using analysis of variance data

$$\frac{\text{Den. (4)}}{\text{Tr.X Den.(24)}} = \frac{7355}{84} = 87.6\ (***)$$

$$\frac{\text{Tr. (6)}}{\text{Tr.X Den. (24)}} = \frac{1222}{84} = 14.6\ (***)$$

$$\frac{\text{Den. (4)}}{\text{Ft.X Den. (8)}} = \frac{7355}{182} = 40.4\ (***)$$

$$\frac{\text{Ft. (2)}}{\text{Ft.X Den. (8)}} = \frac{5751}{182} = 31.6\ (***)$$

$$\frac{\text{Ft. (2)}}{\text{Tr.X Ft. (12)}} = \frac{5751}{76} = 75.7\ (***)$$

$$\frac{\text{Tr. (6)}}{\text{Tr.X Ft. (12)}} = \frac{1222}{76} = 16.0\ (***)$$

*	= P = 0.05	= significant
**	= P = 0.01	= highly significant
***	= P = 0.001	= very highly significant.

sulfur requires nearly 30 days to be stabilized mechanically, but results with dicyclopentadiene and styrene-modified impregnant (20%, based on sulfur weight) indicated that at least 360 days are required for stabilization of mechanical properties. After stabilization the MOE and MOR of the composite made with these same modified impregnants (30%, based on sulfur weight) after

Table 6. Master table analysis of variance MOR of specimens tested after 60 days aging

Source of variation		Gross SS-CT	Net SS	df	Mean sq. variance	F ratio	Sig.
Total	G	198833	14770	104			
	–CT	184063					
Treat-	G	184933	870	6	145	51.8	***
ment	–CT	184063					
Density	G	192933	8870	4	2218	792.1	***
	–CT	184063					
Fiber	G	188155	4092	2	2046	730.7	***
Type	–CT	184063					
Tr.x Den.	G	194404	601	24	25	8.9	***
	–CT	184063					
–NSS	Tr.	870					
–Net SS	Den.	8870					
Ft.x Den	G	197119	94	8	118	4.2	***
	–CT	184063					
–Net SS	FT	4092					
–Net SS	Den.	8870					
Tr.x Ft.	G	189134	109	12	9	3.2	***
	–GT	184063					
–Net SS	Tr.	870					
–Net SS	Ft.	4092					
Error by subtraction							

Includes: Tr. x Den. x Ft.

Separate F test using analysis of variance data

$$\frac{\text{Den. (4)}}{\text{Tr.X Den. (24)}} = \frac{2218}{25} = 88.7\ (***)$$

$$\frac{\text{Tr. (6)}}{\text{Tr.X Den. (24)}} = \frac{145}{25} = 5.8\ (***)$$

$$\frac{\text{Den. (4)}}{\text{Ft.X Den. (8)}} = \frac{2218}{11.8} = 188.0\ (***)$$

$$\frac{\text{Ft. (2)}}{\text{Ft.X Den. (8)}} = \frac{2046}{11.8} = 173.4\ (***)$$

$$\frac{\text{Ft. (2)}}{\text{Tr.X Ft. (12)}} = \frac{2046}{9} = 227.3\ (***)$$

$$\frac{\text{Tr. (6)}}{\text{Tr.X Ft. (12)}} = \frac{145}{9} = 16.1\ (***)$$

* = P = 0.05 = significant
** = P = 0.01 = highly significant
*** = P = 0.001 = very highly significant.

360 days aging were almost the same as those of the composite made with pure sulfur.

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