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Performance of Hybrid Reinforcements in PVC Composites: IV. Use of Surface-Modified Glass Fiber and Different Cellulosic Materials as Reinforcements

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The influence of the composition of various cellulosic materials to surface-treated glass fiber on the mechanical properties of PVC composites have been evaluated. In order to improve the adhesion of cellulosic materials to glass fiber and PVC, celluloses underwent various surface treatments. The properties of the composites filled with glass fiber only are superior to those of celluloses-filled composites. Except impact strength of glass fiber-filled composites and modulus of the composites, in general, the properties are inferior with respect to unfilled polymer. Properties of the hybrid composites comprising celluloses and glass fiber, and composites containing only celluloses enhanced only when the cellulosic materials were surface modified. The compositions of celluloses and glass fibers, where properties improved most, varied with the nature of cellulosic materials and their surface treatments.

KEY WORDS PVC, composites, glass fibers, celluloses

INTRODUCTION

Recent developments in various material applications require inexpensive compounds (e.g., raw materials, and in fabrication) with good properties.¹ The incorporation of high-density inorganic reinforcements, e.g., glass fiber or mica, in a thermoplastic offers a wide variety of property changes, but their use may not result in cost savings on a volumetric basis.^{2–4} The specific gravity of wood flour or cellulosic fibers is roughly 1.5 compared to about 2.5–2.9 for other reinforcements.^{2,5} However, it would seem possible with hybrid composites to exercise greater control over specific properties, thus achieving a more favourable balance between the advantages and disadvantages inherent in any composite material.^{6–8} Therefore, hybridization of conventional reinforcements with cellulosic reinforcements in a common thermoplastic provides another dimension to the potential versatility of reinforced composite materials.^{9–17}

Of course, cellulosic materials present some disadvantages, such as incompatibility with non-polar substances, water absorbancy and thermal instability, which

can be overcome to some extent by proper surface coating.¹⁸⁻²⁰ Moreover, when the mechanical properties of the inorganic reinforcements, such as glass fibers, are compared with cellulosic reinforcements at the same density level (i.e., specific properties), the values obtained for the latter resemble those of the former.²¹

In the present study, the hybrid behavior of surface-treated glass fibers and different cellulosic materials in PVC composites was investigated by evaluating the mechanical properties of the composites. CTMP was surface modified by the pre-treatment of the fiber with maleic anhydride (MA), mixtures of MA and Na-silicate and isocyanate.

MATERIALS

Thermoplastic

PVC-Goodrich (Geon 110 × 334) was supplied by B. F. Goodrich Geon Vinyl Division, Cleveland, Ohio, USA. Before mixing with the reinforcements, PVC was treated with 10% plasticizer, dioctyl phthalate (DOP), and 1.3% stabilizer, barium acetate (BaAc).

Reinforcements

a) Sawdust (SD): Two different varieties of wood species, namely hardwood species aspen (SDA) and softwood species mixtures (75% black spruce and 25% fir) (SDS) were used in the form of wood flour (sawdust). b) Bagasse (BS): The residue of sugarcane bagasse, after extraction of furfural, was supplied by Stake Tech., Co. of Norval, Ontario, Canada. c) Nutshell (NS): Blends of peanut hull and pecan shell of mesh size 100 was supplied by Southeastern Reduction Co., Valdosta, Georgia, USA. d) Glass fiber: Fiber Glass of Canada via Mica Chemical, Montréal. The length of fibers varied from 0.75 to 0.86 mm, and the diameter from 0.014 to 0.0144 mm ($l/d = 52$ to 61.4).

Wood chips for making sawdust and bagasse were ground to a mesh size 60 mixture (60.5%, mesh 60; 20.2%, mesh 80; 15.5%, mesh 100; and 2.5%, mesh 200) with the help of a Granu Grinder (C. W. Brabender Instruments Inc., USA). Cellulosic materials were oven-dried by circulating air at 55°C for a few days.

Coupling Agents, Initiator and Additive

Poly[methylene(polyphenyl isocyanate)] (PMPPIC) was supplied by Polysciences Inc., USA. Maleic anhydride (MA), dicumyl peroxide (DCP), and Na-silicate (NaSc) were supplied by Anachemia, Montréal, Canada.

EXPERIMENTAL

Coating Treatment

To obtain a homogeneous coating, PVC (5 or 10% by weight of cellulose) was used as a coating component. Three different coating compositions were used: a)

PVC (5%) + MA (3% by weight of cellulose) + DCP (1% by weight of cellulose); b) (a) + NaSc (5% by weight of cellulose); c) PVC (10%) + PMPPIC (8% by weight of cellulose). CTMP was mixed with the coating components [e.g. (a) or (b) or (c)], first mechanically at room temperature, then repeatedly (5 to 7 times) with a laboratory roll mill (C. W. Brabender, Model No. 065) at 170°C. After mixing, the coated cellulosic materials were allowed to cool to room temperature, and then ground to mesh size 20.

Preparation of Composites

Mixtures containing 25 g of polymer and coated/non-coated cellulose (25% by weight of composites) were mixed in the roll mill at 170°C. After mixing 5–10 times, the mixtures were allowed to cool to room temperature, and were then ground once more to mesh size 20. The mixtures were then molded into shoulder-shaped test specimens (ASTM D-638, Type V). The mold was heated at 165°C in a Carver Laboratory Press for 10 min under 4.34 MPa pressure and then cooled down to a temperature of ~35°C by circulating cold water for about 10 min in the press. The width and thickness of each specimen were measured with the help of a micrometer.

Mechanical Tests

The mechanical properties [e.g. ultimate tensile strength, ultimate elongation, tensile toughness (fracture energy ÷ volume) and Young's modulus] of all the samples were measured with an Instron Tester (Model 4201) following ASTM D-638. The mechanical properties were automatically calculated with a HP-86B computer. The strain rate was 2 mm/min. Un-notched Izod impact strength was tested following ASTM D-256 with an Impact Tester (Model TMI, No. 43-01) supplied by Testing Machines Inc., Amityville, New York, USA. For impact testing, un-notched specimens were clamped in a sample vise with the bulk of the sample surface subjected to the direction of impact. A pendulum was raised to a fixed height, and then released to break the sample. The energy required to break the specimen was calculated from the difference in pendulum height at the beginning and end of the follow-through swing. The impact strength was expressed as energy/thickness (J/m). The samples were tested after conditioning at $23 \pm 0.5^\circ\text{C}$ and 50% relative humidity for at least 18 h in a controlled atmosphere. The statistical average of the measurements on at least 5 specimens was taken to obtain a reliable average and standard deviation. The coefficients of variation for the mechanical properties varied from 2.5 to 8.5%.

RESULTS AND DISCUSSION

The influence of the composition of various cellulosic materials to surface-treated glass fiber on the mechanical properties of PVC composites appears in Tables I–V. In order to improve the adhesion of cellulosic materials to glass fiber and PVC, cellulose underwent various surface treatments. The effect of such treatment

TABLE I

Tensile strength of glass fiber/cellulosic reinforcements-PVC composites under different coating treatments of cellulotics

Composition of reinforcements (wt%)		Ultimate tensile strength (MPa)			
Cellulosic	Glass	Sawdust (aspen)	Sawdust (spruce)	Nutshell	Bagasse
		49.2 ^a (1.77) ^b	48.1 ^c (1.82)		
Non-treated cellulotics					
100	0	24.2 (2.30)	23.3 (2.38)	28.2 (2.25)	28.1 (1.51)
75	25	25.6 (1.39)	29.6 (1.45)	26.5 (1.81)	27.1 (1.08)
50	50	24.8 (2.50)	29.7 (0.89)	26.6 (1.48)	22.0 (0.71)
25	75	27.8 (0.97)	31.3 (1.76)	28.6 (1.17)	30.8 (0.99)
0	100	34.8 ^d (1.36)			
Cellulotics coated with 3% MA + 1% DCP + 5% PVC					
100	0	26.0 (0.70)	25.6 (1.52)	29.1 (1.63)	30.1 (1.70)
75	25	32.2 (0.76)	30.3 (0.41)	27.3 (1.81)	29.5 (0.92)
50	50	27.2 (1.45)	36.0 (1.75)	27.0 (1.48)	31.8 (1.56)
25	75	28.9 (0.75)	33.7 (1.16)	29.9 (1.17)	31.4 (1.00)
Cellulotics coated with 3% MA + 1% DCP + 5% Na-silicate + 5% PVC					
100	0	25.3 (1.35)	31.5 (0.47)	31.6 (2.65)	31.2 (1.23)
75	25	25.1 (1.44)	31.8 (0.90)	28.6 (1.42)	30.4 (1.40)
50	50	23.8 (1.56)	33.6 (1.86)	25.0 (1.55)	30.4 (1.63)
25	75	29.6 (1.05)	31.7 (1.73)	27.3 (1.31)	28.7 (1.56)
Cellulotics coated with 8% PMPPIC + 10% PVC					
100	0	45.9 (1.17)	47.6 (1.93)	38.1 (2.29)	44.0 (2.27)
75	25	36.5 (1.13)	36.7 (0.87)	38.8 (0.70)	39.2 (1.91)
50	50	32.4 (1.52)	40.0 (1.71)	30.0 (1.85)	36.0 (1.16)
25	75	34.2 (1.63)	34.1 (1.72)	33.2 (0.67)	37.5 (1.87)

^aPVC-virgin. ^bValues in the parenthesis represent standard deviation.

^cPVC + 10% DOP + 1.3% BaAc. ^dMica-filled composite.

on the properties of the composites was also registered in Tables I–V in which the properties of the composites filled with glass fiber only were superior to those of cellulotics-filled composites. Except impact strength of glass fiber-filled composites and modulus of the composites, in general, the properties were inferior with respect to unfilled polymer. Properties of the hybrid composites comprising cellulotics and glass fiber, and composites containing only cellulotics enhanced when cellulotics were surface modified.

Table I reveals that ultimate tensile strength of PMPPIC-coated cellulotics improved most when cellulotics were used alone or along with glass fiber, while MA-coated cellulotics ranked better. When one compared the performance of different

TABLE II

Impact strength of glass fiber/cellulosic reinforcements-PVC composites under different coating treatments of cellulose

Composition of reinforcements (wt%)		Izod impact strength (J/m)			
Cellulosic	Glass	Sawdust (aspen)	Sawdust (spruce)	Nutshell	Bagasse
		10.2 ^a (0.59) ^b	9.9 ^c (0.69)		
Non-treated cellulose					
100	0	7.1 (1.03)	7.2 (1.07)	6.8 (0.71)	6.1 (0.57)
75	25	7.0 (1.18)	7.0 (0.87)	5.9 (0.08)	7.0 (0.89)
50	50	7.2 (1.10)	7.3 (1.12)	6.8 (0.11)	6.2 (0.61)
25	75	7.9 (1.06)	8.2 (0.68)	7.9 (1.11)	7.9 (1.16)
0	100	11.0 ^d (0.45)			
Cellulose coated with 3% MA + 1% DCP + 5% PVC					
100	0	9.1 (1.16)	7.8 (0.47)	7.3 (0.54)	8.1 (1.17)
75	25	7.3 (1.18)	8.1 (0.89)	7.6 (0.82)	9.1 (1.01)
50	50	7.4 (0.23)	8.3 (1.18)	9.8 (0.45)	7.6 (0.03)
25	75	8.5 (0.76)	9.6 (0.96)	10.0 (0.69)	8.3 (1.22)
Cellulose coated with 3% MA + 1% DCP + 5% Na-silicate + 5% PVC					
100	0	7.3 (0.52)	8.3 (1.26)	9.2 (0.38)	8.4 (0.68)
75	25	7.2 (1.07)	8.1 (1.04)	7.6 (0.97)	9.1 (0.48)
50	50	7.7 (1.21)	7.4 (0.46)	6.9 (0.61)	7.1 (0.57)
25	75	8.4 (0.52)	8.1 (0.64)	8.8 (0.68)	8.1 (0.90)
Cellulose coated with 8% PMPPIC + 10% PVC					
100	0	9.5 (1.04)	8.9 (1.22)	9.2 (1.08)	6.7 (0.47)
75	25	8.1 (1.05)	8.0 (1.24)	8.3 (0.54)	7.1 (0.88)
50	50	8.5 (1.25)	8.1 (0.87)	7.8 (0.28)	8.8 (0.88)
25	75	10.2 (0.83)	7.4 (0.60)	9.1 (0.84)	8.6 (0.91)

^aPVC-virgin. ^bValues in the parenthesis represent standard deviation.

^cPVC + 10% DOP + 1.3% BaAc. ^dMica-filled composite.

cellulose with regard to tensile strength of the composites efficiency decreased as follows: sawdust (spruce) > bagasse > nutshell > sawdust (aspen). Table I also revealed that the optimum compositions of cellulose and glass fibers depend on the nature of the cellulose and their prior treatment.

The impact strength of the composites appears in Table II. It is obvious from this table that the impact strength of the composites, particularly when 1:3 (weight ratio) of PMPPIC-treated sawdust (aspen), MA-treated nutshell, and glass fibers are used, exceeds even that of unfilled PVC. For sawdust (aspen), PMPPIC treatment ranked best, followed by MA and MA + silicate treatments. For sawdust (spruce), 1:3 (weight ratio) of MA-treated wood fiber to glass fibers ranked best,

TABLE III

Elongation of glass fiber/cellulosic reinforcements-PVC composites under different coating treatments of cellulосics

Composition of reinforcements (wt%)		Ultimate elongation (%)			
Cellulosic	Glass	Sawdust (aspen)	Sawdust (spruce)	Nutshell	Bagasse
		4.9 ^a (0.24) ^b	4.1 ^c (0.17)		
Non-treated cellulосics					
100	0	1.6 (0.14)	1.5 (0.17)	1.7 (0.23)	1.6 (0.22)
75	25	1.5 (0.12)	1.7 (0.18)	1.8 (0.11)	1.6 (0.16)
50	50	1.6 (0.12)	1.9 (0.08)	1.8 (0.16)	1.4 (0.16)
25	75	1.8 (0.11)	2.0 (0.12)	1.9 (0.17)	1.9 (0.09)
0	100	2.8 ^d (0.07)			
Cellulosics coated with 3% MA + 1% DCP + 5% PVC					
100	0	1.7 (0.17)	1.6 (0.07)	1.9 (0.18)	1.7 (0.15)
75	25	2.1 (0.24)	1.8 (0.07)	1.7 (0.24)	1.8 (0.09)
50	50	1.7 (0.12)	2.1 (0.18)	1.8 (0.16)	2.1 (0.08)
25	75	1.9 (0.12)	1.9 (0.10)	1.9 (0.10)	2.2 (0.12)
Cellulosics coated with 3% MA + 1% DCP + 5% Na-silicate + 5% PVC					
100	0	1.6 (0.06)	1.6 (0.04)	1.9 (0.17)	1.7 (0.09)
75	25	1.5 (0.10)	2.1 (0.08)	1.9 (0.24)	1.9 (0.23)
50	50	1.7 (0.12)	2.1 (0.11)	1.7 (0.13)	2.0 (0.21)
25	75	2.0 (0.09)	1.8 (0.13)	2.1 (0.12)	1.9 (0.09)
Cellulosics coated with 8% PMPPIC + 10% PVC					
100	0	2.7 (0.10)	2.7 (0.25)	2.4 (0.19)	2.5 (0.15)
75	25	2.1 (0.09)	2.2 (0.28)	2.1 (0.08)	2.2 (0.11)
50	50	2.2 (0.14)	2.5 (0.15)	2.4 (0.12)	2.3 (0.16)
25	75	2.1 (0.11)	2.1 (0.12)	2.3 (0.17)	2.3 (0.13)

^aPVC-virgin. ^bValues in the parenthesis represent standard deviation.

^cPVC + 10% DOP + 1.3% BaAc. ^dMica-filled composite.

while MA + silicate and PMPPIC treatments exhibited a similar behavior. On the other hand, for nutshell 1:3 (weight ratio) of MA-treated nutshell and glass fiber seemed best, followed by PMPPIC and MA + silicate. For bagasse, 3:1 (weight ratio) of MA- and MA + silicate-treated bagasse, and glass fibers showed a more or less similar behavior, while PMPPIC treatments had a performance inferior to the other two treatments. However, impact property improved most for sawdust (aspen) and nutshell, followed by sawdust (spruce) and bagasse.

The ultimate elongation of the composites is presented in Table III. Compared to untreated cellulосics fiber-filled composites, elongation improved most when PMPPIC-treated cellulосics alone or their hybrid combination with glass fibers were

TABLE IV

Toughness of glass fiber/cellulosic reinforcements-PVC composites under different coating treatments of cellulose

Composition of reinforcements (wt%)		Tensile toughness (MPa)			
Cellulosic	Glass	Sawdust (aspen)	Sawdust (spruce)	Nutshell	Bagasse
		1.26 ^a (0.10) ^b	0.96 ^c (0.09)		
Non-treated cellulose					
100	0	0.20 (0.03)	0.19 (0.03)	0.23 (0.03)	0.24 (0.03)
75	25	0.23 (0.02)	0.29 (0.02)	0.25 (0.02)	0.23 (0.02)
50	50	0.23 (0.02)	0.33 (0.02)	0.28 (0.03)	0.26 (0.01)
25	75	0.30 (0.02)	0.34 (0.04)	0.27 (0.01)	0.37 (0.03)
0	100	0.53 ^d (0.03)			
Cellulose coated with 3% MA + 1% DCP + 5% PVC					
100	0	0.24 (0.33)	0.22 (0.21)	0.31 (0.04)	0.25 (0.04)
75	25	0.36 (0.03)	0.31 (0.02)	0.28 (0.03)	0.29 (0.02)
50	50	0.24 (0.03)	0.43 (0.03)	0.34 (0.02)	0.36 (0.02)
25	75	0.33 (0.03)	0.33 (0.02)	0.40 (0.02)	0.42 (0.03)
Cellulose coated with 3% MA + 1% DCP + 5% Na-silicate + 5% PVC					
100	0	0.34 (0.04)	0.26 (0.02)	0.29 (0.04)	0.26 (0.03)
75	25	0.31 (0.04)	0.38 (0.02)	0.27 (0.03)	0.35 (0.03)
50	50	0.29 (0.02)	0.44 (0.03)	0.32 (0.03)	0.35 (0.03)
25	75	0.34 (0.02)	0.36 (0.03)	0.30 (0.03)	0.31 (0.02)
Cellulose coated with 8% FMPIC + 10% PVC					
100	0	0.66 (0.03)	0.73 (0.03)	0.54 (0.03)	0.57 (0.04)
75	25	0.40 (0.03)	0.47 (0.04)	0.57 (0.04)	0.48 (0.04)
50	50	0.44 (0.02)	0.59 (0.03)	0.48 (0.02)	0.50 (0.03)
25	75	0.41 (0.03)	0.41 (0.02)	0.47 (0.03)	0.51 (0.01)

^aPVC-virgin. ^bValues in the parenthesis represent standard deviation.^cPVC + 10% DOP + 1.3% BaAc. ^dMica-filled composite.

used. On the other hand, MA and MA + silicate treatments showed more or less the same results. With regard to the performance of different cellulosic materials, their relative efficiencies were: sawdust (spruce) > bagasse ≈ nutshell > sawdust (aspen). Concerning the composition of cellulose and glass fibers, where properties improved most, they varied with the nature of cellulose and their surface treatments.

The tensile toughness (fracture energy ÷ volume) of the composites is shown in Table IV. Toughness followed more or less the same behavior as elongation did (except for the relative performance of nutshell, which was better than that of bagasse). Table V reveals that the modulus of the composites filled with nontreated

TABLE V

Modulus of glass fiber/cellulosic reinforcements-PVC composites under different coating treatments of cellulotics

Composition of reinforcements (wt%)		Young's modulus (GPa)			
Cellulosic	Glass	Sawdust (aspen)	Sawdust (spruce)	Nutshell	Bagasse
		2.0 ^a (0.05) ^b	2.0 ^c (0.18)		
Non-treated cellulotics					
100	0	2.1 (0.19)	2.2 (0.14)	2.1 (0.10)	2.0 (0.18)
75	25	2.3 (0.06)	2.3 (0.19)	2.1 (0.09)	2.3 (0.13)
50	50	2.3 (0.20)	2.3 (0.81)	2.2 (0.10)	2.3 (0.11)
25	75	2.4 (0.08)	2.4 (0.13)	2.2 (0.13)	2.4 (0.13)
0	100	2.5 ^d (0.18)			
Cellulotics coated with 3% MA + 1% DCP + 5% PVC					
100	0	2.3 (0.17)	2.5 (0.12)	2.3 (0.11)	2.3 (0.10)
75	25	2.3 (0.10)	2.3 (0.11)	2.1 (0.09)	2.3 (0.08)
50	50	2.5 (0.11)	2.5 (0.10)	2.1 (0.16)	2.3 (0.11)
25	75	2.5 (0.10)	2.4 (0.12)	2.4 (0.10)	2.5 (0.09)
Cellulotics coated with 3% MA + 1% DCP + 5% Na-silicate + 5% PVC					
100	0	2.3 (0.20)	2.7 (0.04)	2.4 (0.14)	2.6 (0.07)
75	25	2.2 (0.03)	2.2 (0.07)	2.1 (0.11)	2.2 (0.14)
50	50	2.5 (0.17)	2.5 (0.14)	2.2 (0.06)	2.3 (0.11)
25	75	2.4 (0.09)	2.4 (0.19)	2.4 (0.11)	2.3 (0.13)
Cellulotics coated with 8% PMPPIC + 10% PVC					
100	0	2.4 (0.10)	2.4 (0.24)	2.3 (0.12)	2.4 (0.18)
75	25	2.2 (0.18)	2.3 (0.18)	2.1 (0.11)	2.4 (0.14)
50	50	2.2 (0.05)	2.3 (0.12)	2.2 (0.20)	2.3 (0.10)
25	75	2.4 (0.11)	2.5 (0.13)	2.4 (0.10)	2.5 (0.17)

^aPVC-virgin. ^bValues in the parenthesis represent standard deviation.

^cPVC + 10% DOP + 1.3% BaAc. ^dMica-filled composite.

cellulotics is inferior to that of only glass fiber-filled composites. But when coated cellulotics were used, the same property enhanced in a similar fashion to that of single glass fiber-filled composites. Furthermore, modulus improved most when the proportion of coated cellulotics was equal or less than that of glass fiber. Sawdust (spruce) ranked best, followed by sawdust (aspen), bagasse and nutshell. With respect to various coating treatments, MA ranked best, while MA + silicate and PMPPIC treatments ranked similar.

The above-mentioned results indicate that surface-treated cellulosic materials offer better mechanical properties when they are used alone or as a hybrid filler with surface-treated glass fibers in PVC composites, in comparison with those of

untreated ones. In fact, prior coating of the cellulose fiber with a thermoplastic (e.g. PVC) produces a soft film of hydrophobic materials on the surface of the hydrophilic cellulose fiber.^{22–24} As a result, the phase separation between the two different matrices may be reduced. In addition, strong fiber-fiber interaction due to intermolecular hydrogen bonding has also been diluted, which leads to a better dispersion of the fibers. Moreover, during the coating of the cellulose fiber with MA and a thermoplastic in the presence of an initiator (e.g. DCP), the polymer (e.g. PVC) and cellulose may link together by means of MA.^{25–29} Moreover, the —OH group of cellulose also has the ability of forming hydrogen bonds, as well as ester linkage with the —COOH group of the MA segments.

In fact, Na-silicate did not offer any chemical interaction with either of the components of the hybrid composites. As a result, the use of silicate with MA did not show any significant influence on the mechanical properties of the composites. But it provided various advantages compared to polymeric coatings. For example, they protected the composites against the effects of moisture because of their dryness and heat resistance coating. Furthermore, coated fiber would have a much lower bulk volume than that in a non-coated state.^{22,30}

It has also been reported in our previous publications that coupling agent PMPPIC, which is an efficient coupling agent, forms a “bridge” between cellulosic fibers and PVC in the interface.^{31,32} Moreover, glass fibers were surface-treated with a silane coupling agent and they were found to be a good reinforcing filler when used alone or along with coated wood fibers in thermoplastics, e.g., polystyrene composites.^{14–17} As a result, the combination of treated cellulose fibers and treated glass fibers seemed a good reinforcing filler for PVC composites.

It is also obvious that the performance of the various cellulosic materials is not the same. In fact, the performance of cellulose, as a reinforcement for plastics, depends on their quality, e.g. fiber length, fiber-making technique, morphology, lignin content, inherent physical and mechanical properties, origin, etc.^{33–35} It is a well-established fact that fiber length is a critical parameter in the evaluation of the composites' properties.³⁶ In the present study, sawdust and bagasse were used with mesh size 60, while nutshell required mesh size 100. Therefore, it is also very difficult to compare the performance of different cellulosic materials with different mesh sizes.

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