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Effect of Precipitated Calcium Carbonate on Physical, Mechanical and Thermal Properties of Cassava Starch Bioplastic Composites

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***Abstract*— The development of bioplastic composites from various natural polymers reinforced with Precipitated Calcium Carbonate (PCC) has become a field of increasing interest. In this study, the effect of PCC on the physical, mechanical and thermal properties of a cassava starch matrix composite was examined. The bioplastic composites were made of cassava starch and mixed with glycerol as a plasticizer and 0-10% by weight of PCC. The material was then poured into a mold and oven dried. The physical, thermal and mechanical properties of bioplastic/PCC composites were investigated using Tensile Strength measurements, X-Ray Diffraction, Thermogravimetric Analysis, Scanning Electron Microscopy (SEM), and Fourier Transform Infrared Spectroscopy (FTIR). The optimum tensile strength was obtained upon the addition of 4 % PCC. The addition of PCC improved the thermal stability of bioplastic/PCC composites. The results of X-ray Diffraction testing showed an increase in the crystallinity of the bioplastic/PCC composites with increase in PCC content but there is a decrease in the moisture absorption. SEM images indicated that the PCC filler content was incorporated into the matrix. In general, FTIR indicated the bioplastic/PCC composites were hydrophilic and the addition of PCC reduced the hydrophilic properties by damaging the hydrogen bonding between starch molecules and water.**

***Keywords*— cassava starch; precipitated calcium carbonate; tensile strength; thermal stability; moisture absorption**

I. INTRODUCTION

Agriculturally produced biopolymers can be used to manufacture bioplastics that can replace synthetic plastics in pharmaceutical and food packaging [1]. Starch is a polysaccharide biopolymer. It is abundant, cheap, and fully biodegradable and forms a matrix that could become a possible replacement of synthetic polymers derived from petroleum without causing environmental pollution. However, starch has some limitations.

Cassava (Manihot esculenta) or tapioca is abundantly and cheaply available in Indonesia making it a candidate for bioplastic manufacture. However, it has inferior mechanical and thermal properties. The fragile nature of cassava starch bioplastic can be overcome by adding plasticizers such as glycerol or sorbitol. These increase flexibility but reduce the strength [3]. As a starch, it is also hydrophilic and absorbs water from the atmosphere in storage [2]. Various efforts have been made to improve the mechanical properties by using natural fibers/biomass as a reinforcement. Natural fibers that have been used as fillers in these biocomposites include pineapple leaf fiber, kenaf fiber, water hyacinth fiber, hemp fiber, oil palm empty fruit bunch fiber, palm leaf fiber and sago fiber [4], [5].

The thermal stability and mechanical properties of biocomposites can also be improved by adding an inorganic filler such as Calcium Carbonate ($CaCO_3$). $CaCO_3$ increases tensile strength, modulus, flexural strength and heat deformation in polypropylene (PP). $CaCO_3$ may also increase fracture resistance in PP [6]. Use of nanoparticle-CaCO3 as a filler in a PP matrix has been shown to improve thermal, rheological, and mechanical properties [7]. Latinwo et al [8] investigated the effects of different particle sizes and compositions of $CaCO_3$ on the mechanical properties of polyurethane foam. Baek et al [9] also conducted research on Poly Lactic Acid matrix bioplastics reinforced with PCC and found particle size of PCC was homogeneously distribution. $CaCO_3$ particles increased the strength and prevented cracks in PLA/PCC composites. This study investigates the impact of a PCC filler on physical, mechanical and thermal properties of cassava starch bioplastic composites.

II. MATERIALS AND METHODS

A. Material

Cassava Starch (Cap Tani, Indonesia), PCC with an average size of 13 μm (Sigma-Aldrich, Germany), and glycerol (PT. Cisadane Raya Chemicals, Tangerang Indonesia) were used for the fabrication of bioplastic/PCC composites.

B. Fabrication of bioplastics/PCC composites

Fabrication of bioplastic/PCC composites was based on a modification of Tongdeesoontorn et al [3]. Cassava starch was dissolved in distilled water (7.14% w/v) and PCC (% w/w from cassava starch) with different concentrations in distilled water (Table 1). Glycerol (25% w/w of cassava starch) was added as a plasticizer. The starch /$CaCO_3$/glycerol solution was heated to 100 ^{0}C with constant stirring (350 rpm) until gelatinization, cast into 20 x 20 x 0.3 cm^3 rectangular glass molds then oven dried at 50^{0}C for 17 h. This process is illustrated in Figure 1.

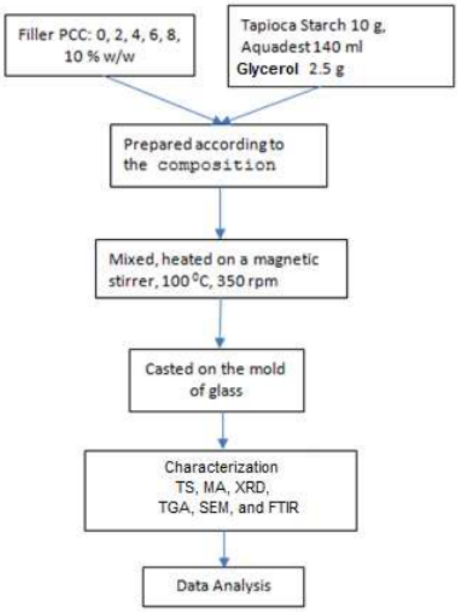


Fig. 1 The process of synthesis of bioplastic/PCC Composites

C. Mechanical Properties

A 95T Series Com-Ten testing machine was used to measure the tensile strength of the composite samples using a speed of 2 mm/min and room temperature. Tensile testing used standard methods of the American Society for Testing and Materials (ASTM). Tensile strength was tested in accordance with the ASTM D 638 Standard Test Method for Mechanical Properties of Plastics with specimen dimension type I with sizes as shown in Table 2

TABLE I:
THE COMPOSITION OF PCC IN FABRICATION BIOPLASTIC/PCC COMPOSITES

Bioplastics/PCC	Composition (g/140 ml distilled water)		
	Cassava starch	Glycerol	PCC
Control/Bioplastics	10	2.5	0
+PCC 2%	10	2.5	0.2
+PCC 4%	10	2.5	0.4
+PCC 6%	10	2.5	0.6
+PCC 8%	10	2.5	0.8
+PCC 10%	10	2.5	1

TABLE II:
DIMENSIONS OF TENSILE TEST SPECIMENS [10]

Dimensions (see drawings)	For thickness 7(0.28) or under, mm (in)		For thickness Over 7 to 14 (0.28 to 0.55), mm (in)
	Type I	Type II	Type III
W-Width of narrow section^{E,F}	13 (0.50)	6 (0.25)	19 (0.75)
L-Length of narrow section	57 (2.25)	57 (2.25)	57 (2.25)
WO-Width overal, min^{G}	19 (0.75)	19 (0.75)	29 (1.13)
LO-Length overal, min^{H}	165 (6.50)	183 (7.20)	50 (2.00)
D-Distance between grips	115 (4.50)	135(5.30)	115 (4.50)
RO-Radius of fillet	76(3.00)	76(3.00)	76(3.00)

D. Moisture absorption bioplastic/PCC Composites

Moisture absorption was measured using a modification of the method used by Abral et al. [11]. 20 x 20 x 0.3 mm^3 samples were dried until constant weight then weighed. They were then exposed to 99% humidity at 25 ± 2 °C. Moisture absorption after time t (%) was calculated to be ((Wt-Wo)/Wt) x 100 %, where Wt is the weight of bioplastic/PCC composites after water absorption and Wo was the initial weight of the dried sample.

E. X-ray Diffraction

The structure of the biocomposites/PCC were studied by X-Ray Diffraction (XRD). The XRD diffractogram was recorded using a series PANalytical's X-ray diffractometer. NI filtered CuKα radiation at wave number 1.54060 A was used with voltages of 40 kV and 30 mA and scan angles 2θ of 2-100 ^{0} every 20 ^{0} / min.

F. Thermogravimetric

TGA4000 (Perkin Elmer) was used to measure the degradation of bioplastic/PCC composites with heating. The heating rate was 10 °C per min from 50 to 400 ^{0}C. Nitrogen flow rate during the trial was 40 ml per min.

G. SEM observation

The surface morphology of biocomposite / PCC was observed using SEM images according to the method of Abral et al. (2014) [11] using a 3400 N series Hitachi SEM.

H. Fourier Transformed Infrared Spectroscopy (FTIR)

FT-IR analysis was performed from wavenumber 400 to 4000 cm^{-1} to detect the presence of functional groups in the bioplastic / PCC composite for each PCC concentration using a Frontier (Perkin Elmer) FT-IR spectrometer.

III. RESULT AND DISCUSSION

A. Mechanical Properties of Bioplastic/PCC composites

The mechanical properties of bioplastic/PCC composites can be seen in Figure 2.

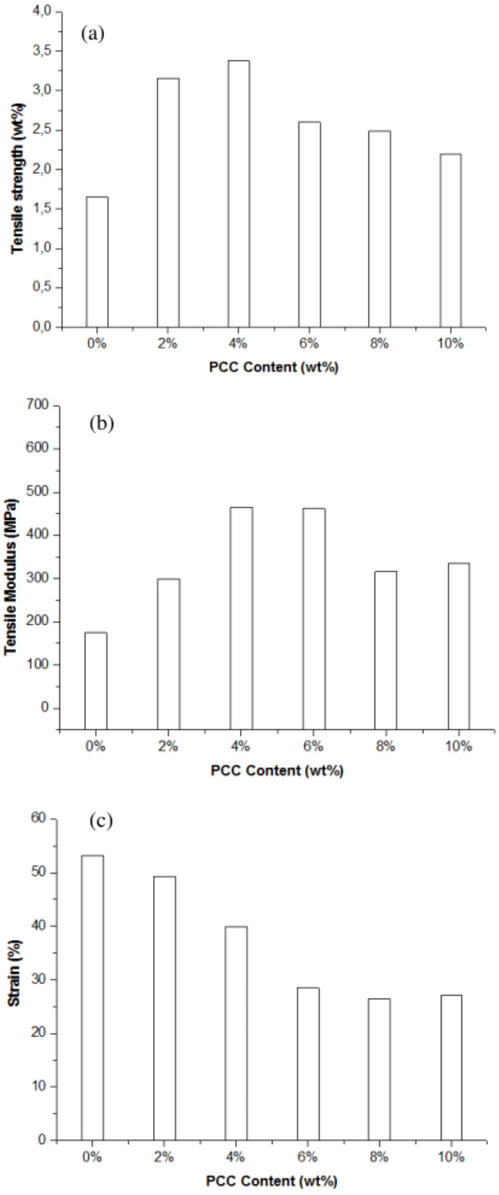


Fig. 2 Effect of PCC on mechanical properties of the composites. (a) tensile strength, (b) tensile modulus and (c) strain

Figure (2 a) shows an increase in tensile strength from 1.65 MPa to 3.38 MPa with the addition of PCC up to 4% (w/w). The modulus of Elasticity (Figure 2 b) increased from 174.61 MPa for pure bioplastic until 645.15 MPa with the addition of 4% PCC.

This increase in tensile strength and modulus of elasticity is related to the hydrogen bonding that occurs between the PCC and the cassava starch matrix. The tensile strength of bioplastic composites is also improved as the small size PCC fills the cavities in the starch matrix during fabrication [12]. Further addition of PCC above 4% reduces tensile strength and modulus of elasticity. This decrease in tensile strength and modulus of elasticity is probably due to the presence of porosities and aggregation which occurs in the matrix as PCC content increases. Wicaksono et al (2013) found that the tensile strength of a cassava starch bioplastic with filler was 1.45 Mpa which increased with the addition 2% and 4 % cellulose nanofibers (CN) to 2.48 MPa and 2.75 MPa, respectively [13]. Saurabh et al (2015) reported that addition of up to 10% nanoclay significantly improved the mechanical properties of nanocomposites however a higher concentration of nano-clay led to a sharp decrease in mechanical properties and caused cracks in the bioplastic composites [14].

The addition of 4% w / w of $CaCO_3$ significantly affected the strain of bioplastic/ PCC composites properties, the strain decreased from 53.14% to 39.91% (Fig. 2c). The same is true for all $CaCO_3$ additions, the smallest strain of 26.50% occurring for the addition of 8% CaCO3. This result can be attributed to reduced interaction between CaCO3 and the tapioca matrix when the excess CaCO3 is unable to be absorbed into the matrix. These changes in mechanical characteristics are similar to those caused by other fillers in composites in previous studies [15].

Mechanical properties of the bioplastic composites are highly dependent on the interfacial interaction between matrix and filler. Increased surface contact allows increased hydrogen bonding between matrix and filler strengthening the composite [16].If the concentration of PCC exceeds 4%, the matrix cannot cover the entire surface of the PCC particles this hydrogen bonding is reduced so strength is reduced also.

B. Moisture Absorption Bioplastic/PCC composites

The highest value for moisture absorption of the pure bioplastic (0% PCC) over 21 was 70.59% (Figure 3). The filler effectively reduces the moisture absorption [17]. The hydrophobic nature of the filler reduces absorption of water vapor in the hydrophilic bioplastic as seen over the 42.5 hours. 10% PCC bioplastic had the lowest (53.33%) moisture absorption at 99% relative humidity. Better distribution throughout the bioplastic/PCC composites and the bonding between matrix and filler also affects the absorption of water vapor.

In general, there are two mechanisms for diffusion of water in a composite like this. The gap between the matrix and filler can become a pathway of diffusion water or the cracks and weaknesses at the interface of the fiber and the polymer matrix can produce capillary action. Water absorbed into the polymer can be free water or bound water. Free water can move through the microvoids and pores, while bound water molecules disperse in the matrix as they are attached to the polar groups of the polymer [18], [19].

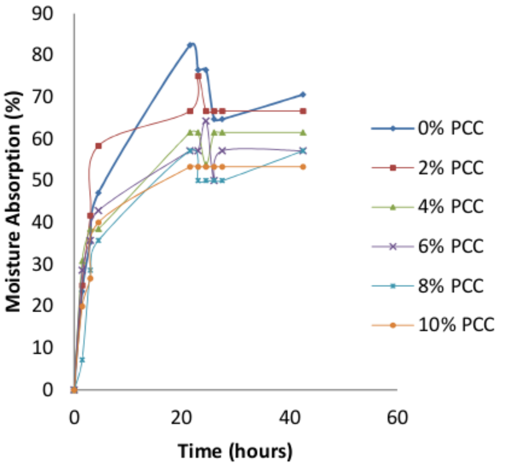


Fig. 3 Moisture absorption of bioplastic/PCC composites

C. X-Ray Diffraction

Figure 4 shows the XRD profile for biocomposites. Based on information from the Joint Committee on Powder Diffraction Standards database shown in Figure 5, the $CaCO_3$ crystal system, is rhombohedral with, a (Å): 4988, b (Å): 4.9880, c (Å): 17 061, and Alpha (^{0}): 90, Beta (^{0}): 90, Gamma (^{0}): 120, while the tapioca matrix cellulose is $C_6H_{10}O_5$which has a monoclinic crystal system, a (Å): 7784, b (Å): 8201, c (Å): 10.380, and Alpha (^{0}): 90, Beta (^{0}): 90, Gamma (^{0}): 96.5.

Starch granules can take amorphous and crystalline forms and are mainly composed of amylose and amylopectin. They are destroyed by heat and shear forces from the mixing during the manufacturing process of the biocomposite resulting in a linear amylose polymer. Thermoplastic starch is characterized by broad peaks at $2\theta = 170$, indicating a fully amorphous matrix [20].

The diffraction pattern of the $CaCO_3$ and tapioca starch films is shown in Fig. 4. The X-ray diffraction pattern of pure tapioca starch films shows low peak intensity. The diffraction peaks are narrower indicating lower crystallinity. The characteristic peaks at 2θ: 17.10, 20.9. and 22.90 are evident. The diffraction pattern is probably due to the strong interaction between the hydroxyl groups of the starch molecules are replaced by hydrogen bonds formed between plasticizer and starch during processing [21].

The characteristic diffraction peaks of biocomposites /PCC was similar to that of the tapioca starch matrix and pure $CaCO_3$. Characteristic peaks can be detected at an angles 2θ: 23^0, 36^0, 39^0, 43^0 and 480 indicating a good compatibility between tapioca starch containing cellulose with calcium carbonate. The narrowness and high intensity of the peak at 2θ: 29.5^0 $CaCO_3$ (41.5^0) shows the high level of calcium carbonate crystallinity.

A widening of the diffraction peaks can be observed in Figure 4, Sun (2014) explains that, according to the kinematical scattering theory, expansion of the X-ray peak indicates imperfections in the crystal lattice or can be due to the small crystal size [21]. As expected, the addition of PCC nanoparticles can modify the peak intensity of the film biocomposites / PCC indicating higher crystallinity hence explaining the superior strength over pure cassava starch.

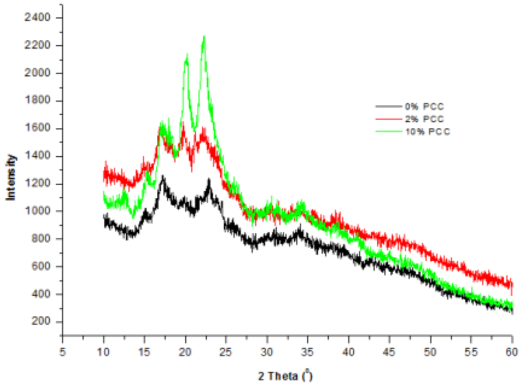


Fig. 4 XRD spectra of bioplastic/PCC composites

D. Thermogravimetric Analysis

TGA was carried out to study the thermal degradation of Bioplastic/ PCC composites. Figure 5 shows the % weight loss of bioplastic/PCC composites vs temperature for several concentrations of PCC. Composite bioplastics degrade between 250 - 300^0C and become stable after 340^0C. The mass loss at temperatures of 50 – 200 ^{0}C was caused by the loss of water. Teixeira et al. (2009) reported that the mass loss of 100 ^{0}C to onset decomposition temperature associated with the evaporation of water [22]. The main mass loss occurs between 250 ^{0}C and 330 ^{0}C, ascribed to the decomposition of the polymeric films [23].

Pure starch bioplastics have a decomposition phase at about 312^0C corresponding to the temperature of the thermal depolymerization of the starch [24]. Bioplastic/PCC composites with a concentration of 10% PCC have the highest thermal stability. The addition of PCC improves thermal stability indicated by the slower weight loss on heating at this decomposition stage.

E. SEM observation

SEM analysis was conducted to determine the composite micro-structure (Figure 6). The pure starch bioplastic surface appears homogeneous and smooth (Fig. 6 a and 6 b). As PCC content increases above 4%, the surface becomes visibly uneven with signs of PCC particle aggregation. This confirms the supposition that the decline in mechanical strength of the biocomposite with a concentration of more than 4 % PCC is related to the extra PPC being unable to be absorbed into the matrix and bonded to the starch polymers. Besides, in Figure 6(c) and 6(d) porosity was found to reduce the tensile strength of bioplastic/PCC composites.

F. Fourier Transformed Infrared Spectroscopy (FTIR)

All FTIR showed a peak was evident at wave number 3200-3600 cm-1 (Figure 7). A peak around 3296 cm-1 indicates a hydrogen bonded O - H stretching [25]. All samples peaks corresponding to a O - H functional groups, but with slightly different wave numbers between 3296 cm-1 and 3295 cm^{-1} depending on the PCC levels. The transmittance intensity of this peak decreases with increased levels of PCC filler above 4%, indicating hydrogen bonds between the starch molecules have been damaged [26].

All bioplastic composite samples had a peak in the range 2800-2950 cm^{-1} corresponding to a C - H functional group [27]. Another peak in the range 1620 - 1650 cm^{-1} corresponds to water absorption. A peak at 1320-1380 cm^{-1} in corresponding to bending vibration of a C - H group and C - O of an aromatic ring was also evident [28,29]. Peaks corresponding to bending vibration of C - O, and O - H in the range of 1010-1070 cm^{-1} was also present in all samples.

Increased levels of PCC filler in bioplastic composites resulted in lower transmittance indicating higher absorption at these wavenumbers. [30]. These results indicate that there have been changes in the starch matrix with the addition of filler chemicals. The interaction between these particles creates hydrogen bonds during processing. These hydrogen bonds strengthen the mechanical properties of the bioplastic composite. Bodirlau et al states that the bonds are probably between hydroxyl and carbonyl groups in the starch and carbonyl and hydroxyl groups and hydroxyl groups in the cellulose [31].

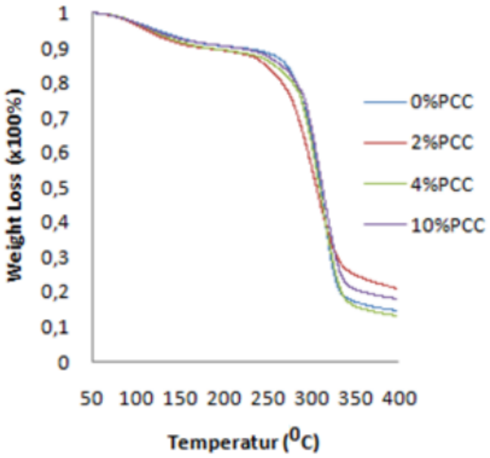
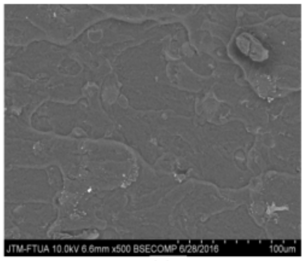
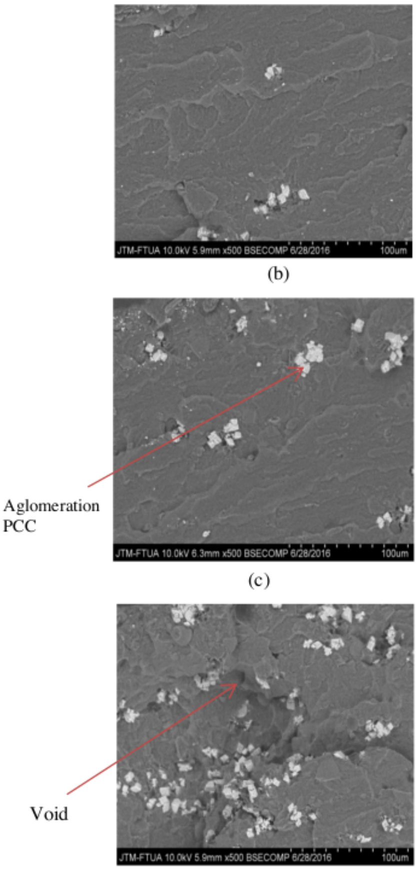


Fig. 5 TGA curves of bioplastic/PCC composites



(a)



(b)

(c)

(d)

Fig. 6 SEM micrographs of bioplastics/PCC composite (a) 0%PCC, (b) 4%PCC, (c) 8% PCC, and (d) 10% PCC

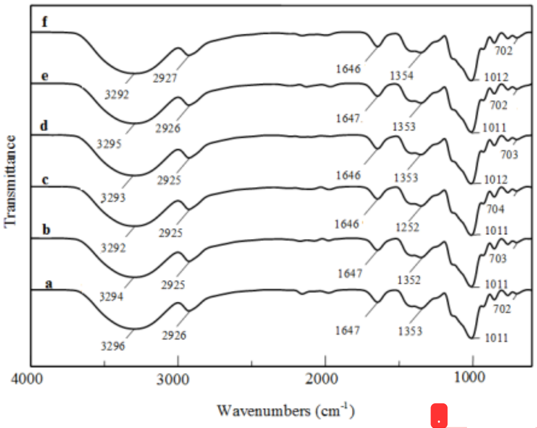


Fig. 7 Infrared spectra of bioplastic/PCCcomposites(a) 0%PCC, (b) 2%PCC, (c) 4% PCC, (d) 6% PCC, (e) 8% PCC and (f) 10% PCC

IV. CONCLUSIONS

The addition of precipitated calcium carbonate (PCC) into a cassava starch matrix increased the tensile strength of the resulting bioplastic/PCC composite with an optimum value with the addition of 4 % (w/w) of PCC. This resulted in a composite with tensile strength and modulus of elasticity of 3.38 MPa and 465.15 MPa, respectively. Analysis of TGA graph shows that the addition of PCC improved the thermal stability of the composite. The moisture absorption test indicated that the composites were hydrophilic, although the water absorption properties were reduced from 70.59 % to 53.33 % at 99 % RH after the addition of 10 % PCC. The XRD results test showed bonding between the tapioca starch cellulose and PCC. After PCC content exceeded 4% distribution was no longer even and aggregation occurred decreasing the tensile strength

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