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Determination of the minimum sample size for a reliable strength measurement of talc-filled polypropylene fibers

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ABSTRACT: Accurate determination of mechanical properties plays an important role to comment on improvement in the mechanical properties of particle-filled PP fibers. However, the existing standards are not totally suitable for reliable strength determination of particle-filled PP fibers. In the framework of this study, microsized talc particle-filled PP fibers were produced with different talc ratio and tensile strength measurements were performed with various gage lengths. Statistical Akaike Information Criterion analysis shows that strength distribution of talc-filled PP fibers is best characterized by Weibull distribution function. It is reported that, the gage length has almost no influence of Weibull parameters of pure PP fibers while strong effects on Weibull parameters of talc-filled PP fibers. It is shown that if the tensile strength of talc-filled PP fibers is to be measured, at least 50 samples, which is more than value suggested by existing standard, should be used for a reliable determination of Weibull parameters. Therefore, the main aim of this study is to question the feasibility of minimum sample size suggested by the existing ASTM D3822 standard for reliable strength measurement of talc filled PP fibers. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 44083.

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INTRODUCTION

Polypropylene (PP) polymers and fibers have high strength, elastic modulus, and chemical resistance. PP fibers are preferred in the textile industry due to their low cost and easy processing ability and low-density.[1] Recently, both unfilled and particle-filled PP polymers were used in increasing number of applications especially in the automotive, electrical, and textile industries.[2,3] Moreover, both PP and particle-filled PP were also preferred in products where creep resistance, high stiffness and high toughness were required.

PP fibers and yarns were used in carpets, underlays, rugs, hygiene textile products, tapes, ropes, clothing, geotextiles, technical, and medical textiles.[4] Increase in the application areas of PP fibers resulted in the need of additional properties and functions. Therefore, it has become necessary and important to produce particle-filled PP fibers and yarns to improve material properties.[1,5–14] Takahashi and Kimura[12] added polydimethylsiloxane and/or oleamide to pure PP fiber and reported an improvement in the wear resistance. Broda *et al.*[13] produced PP/stearic acid composite fibers and observed higher linear density, higher elongation at break, and slightly lower tenacity when compared to fibers produced from pure PP. Jeong *et al.*[14] mixed PP with the nano-sized silver particles to provide an antibacterial property.

Several articles[15–23] have pointed out the scattering in strength and dependence of the strength on specimen size (e.g., diameter, length) of various fibers used in textile applications. It has been reported that strength of natural fibers is a function of fiber diameter and the length of the fiber and there is no specific value to represent deterministically their strength. This leads to the necessity of employing an efficient method for the evaluation of the fiber strength and the prediction of its size dependence.[15–18] The statistical variability in single fiber strength of natural fibers was observed due to the randomly distributed flaws along the fibers and variability in the fiber diameter. In contrast to natural fibers, investigations with aramid and Kevlar fibers showed that the gage length has little effect on fiber strength.[19–21] Lim *et al.*[21] performed axial tension and transverse compression experiments on single fibers (Kevlar®, Kevlar® 129, and Twaron®) with diameters in the order of 9–12 μm. Results showed that gage length and strain rate have little effect on the tensile strength of PPTA single fibers. Studies with high strength polymer fibers show a discrepancy whether there is a size effect or not.[22,23] Schwartz *et al.*[22] observed no influence of strain rate and gage length (10–200 mm) on strength of ultra-high strength polyethylene fibers. On the other hand, Naito[23] obtained scatter in strength of high strength polyethylene fibers.

In the studies described earlier,[15–29] the fibers and yarns were produced from polymers without any filling materials. In recent years, the addition of functionality to polymeric fibers, different types of filling particles and/or polymers are mixed with the polymer matrix during the fiber production. Therefore, reliable strength characterization of particle-filled fibers becomes very important. In this study, the strength behavior of talc-filled PP fibers was investigated. The main motivation of adding talc particles to PP fibers is to increase the thermal shock resistance and thermal stability[24] of the pure-PP fibers. Incorporating talc in a PP material increased the thermal conductivity, resulting in a faster heating and cooling rate, and thermal shock resistance.[24] In addition, Tascan and Nohut[24] showed that an addition of talc particles into pure-PP causes the fiber to behave in a more brittle nature. In general, brittleness in materials introduces a size effect (decrease of strength with increasing the specimen size) and scatter in strength.[25] Due to this so-called size effect, the effect of gage length on the mechanical properties of talc filled PP fibers is expected to play a critical role in reliable strength determination. Furthermore, since the positions of the talc particles in the fiber become the origins of the failure points of the fibers, it is expected that the strength of talc-filled fibers will scatter and therefore statistical methods should be used in the analysis.[25] The main aim of this study is to investigate the influence of gage length and sample size (the number of the samples used in testing) on the mechanical properties of talc-filled PP fibers

In this article, firstly the most suitable fitting function to characterize the strength of talc-filled PP fibers is selected from the three most versatile distribution functions (i.e., normal, log–normal, and Weibull distribution functions) by using Akaike Information Criterion (AIC). All the single fiber strength could be measured according to ASTM D3822.[26] In this standard, at least 20 random specimens from each laboratory sampling unit should be tested to measure the tensile properties of single textile fibers. As addition of talc particles may result in a different fiber structure, increase in brittleness and scatter of strength, it should also be questioned whether 20 specimens is enough to measure the tensile properties of talc-filled PP fibers in a reliable manner or not. In the final part of this article, the minimum number of tests for reliable strength result will be determined. Therefore, the main aim of this study is to question the feasibility of minimum sample size suggested by the existing ASTM D3822 standard for reliable strength measurement of talc filled PP fibers.

EXPERIMENTAL

Materials

PP chips (density at 23 °C is 0.9 g/cm^3) that contain different percentages of microsized (1–3 microns) talc (i.e., 10%, 18%, and 32%) particles (GEMAPILEN BKT Series) were supplied from GEMA Polymer Company. During the production of talc filled PP chips, the round-shaped talc particles were added to PP chips before it is fed into the injection molding or extrusion machine via the use of a dosing or mixing unit in the same way as a traditional master batch would be. It is then transported into the manufacturing process.

PP is mixed with microsized talc particles to produce mixed fiber to increase thermal shock resistance of the fiber.[24] Viscosity of the melted mixture plays a very important role in the spin ability of the fibers and yarns. The amount of talc particles affects the viscosity since these particles will be in solid form during the production of fibers and yarns. 50% of talc added PP mixtures and 50% of 25 mfi 100% Capilene PP chips, provided from Carmel Olefins Company, were mixed at different amounts to achieve same viscosities with different talc percentages. The mixed chips were then put into the feeder of the spinning unit. These different percentages of talc filled PP chips were then used to produce fibers using a typical pilot melt spinning unit at Zirve University Fiber Production Center (see Figure 1).

All fiber samples were produced at standard room conditions (i.e., at 20–23 °C and at 50% humidity). At the melt spinning unit, spinneret with 16 holes, each has 500 µm diameter was used.[24] The spun fibers were fast cooled with cool air and drawn with drawing ratios of 8.33 at the first drafting region and 1.8 at the second drafting region. With the given drawing ratio, different amounts (0%, 5%, 10%, and 15%) of talc filled PP fibers have been produced. Optical microscope images of produced fibers with different talc ratios are shown in Figure 2. The diameter of fibers at the same amount of talc addition was observed to be consistent.

Experimental Setup

Quasi-static tensile mechanical properties of talc filled fibers were measured according to ASTM D3822 standard at atmospheric room conditions 22 °C and at 65% humidity using an INSTRON@ 5944 tensile tester equipped with a load cell of 5 kN.[27] The standard suggests nominal gage length of at least 10 mm and when applicable 250 mm. The rate of extension in the tests was adjusted to 60% of the initial specimen length/min. In the framework of this study, from each type of PP fiber containing different amounts of talc particles (i.e., 15%, 10%, 5%, and pure PP), 55 samples have been used for the tensile testing with gage lengths of 12.5, 25, 50, 75, and 100 mm.

Optical microscope images of the microstructure of the samples were taken by using Olympus CX25 microscope with 5MP camera. The ASTM D3822[26] standard suggests testing of minimum 20 random specimens for the determination of tensile properties. More than one component in the fiber may change the whole microstructure therefore the strength behavior of the fiber. ASTM D3822 standard does not specify minimum specimen size regarding tensile property measurements for particle-filled textile fibers. Therefore, testing the strength of 20 particle-filled textile fibers should be questioned whether it provides trustable results or not.

Theory/Calculation

Akaike Information Criterion. Akaike information criterion (AIC) was used to determine the best fitting function. AIC is based on the similar consideration as the log-likelihood ratio

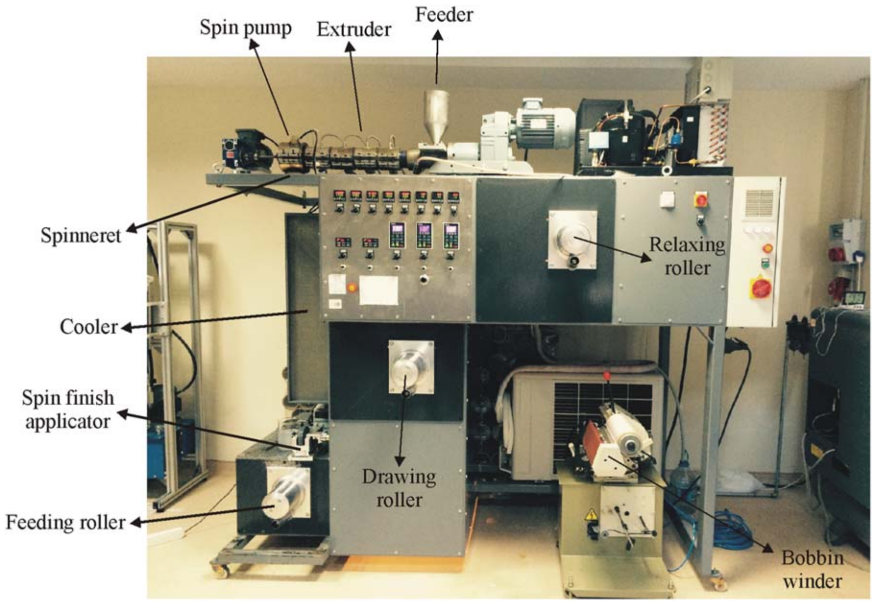

Figure 1. Pilot melt spinning unit at Zirve University Fiber Production Center. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and is a more promising method to obtain the confidence bounds and measures the goodness-of-fit of an estimated statistical model by linking the likelihood to a distance between true (experimental) and assumed distributions.[28] The AIC index, which has been used in a number of areas as an aid to select between competing models, is defined as

$$\mathrm{AIC} = -2\ln \hat{L} + 2k \tag{1}$$

where k is the number of parameters to be fitted (for example, $k=2$ for a two-parameter Weibull distribution), $\ln \hat{L}$ is the maximized log-likelihood for a given model and can be calculated by:

$$\ln L = \sum_{i=1}^{n} \ln f(Y_i) \tag{2}$$

where n is the number of data and $f(Y_i)$ is the probability density function (pdf) of an estimated distribution. The AIC values can be directly compared, preferring the distribution which gives the smallest value and the difference in values Δ(AIC) corresponds to a reliable indication that one distribution is superior to another.[29]

Distribution Functions. Weibull distribution function has been mostly used in the characterization of the tensile strength of brittle fibers and ductile polymer textile fibers and yarns.[23,30–38] The main reason of using Weibull distribution is that Weibull modulus can be a useful tool to evaluate the fiber's homogeneity in terms of properties and it is a good quality control parameter during processing. The simplest form of a Weibull function can be written as.[39,40]

$$P(\sigma, V) = 1-\exp\left[-\frac{V}{V_0}\left(\frac{\sigma}{\sigma_0}\right)^{m}\right] \tag{3}$$

where $P(\sigma, V)$ is the cumulative failure probability of a component due to volume flaws, V is the volume of the component, V_0 is the unit volume, σ is the uniaxial applied stress, m is the Weibull modulus which describes the scatter of strength, and σ_0 is the characteristic stress at which the failure probability is

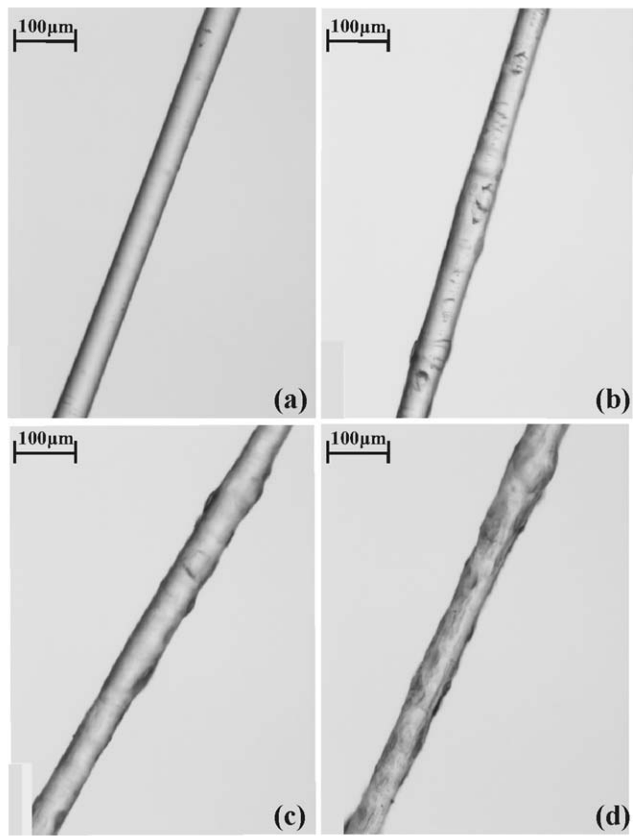

Figure 2. Optical microscope images of talc-filled PP fibers with talc ratios of (a) 100% PP, (b) 5% talc-filled PP, (c) 10% talc-filled PP, and (d) 15% talc-filled PP.

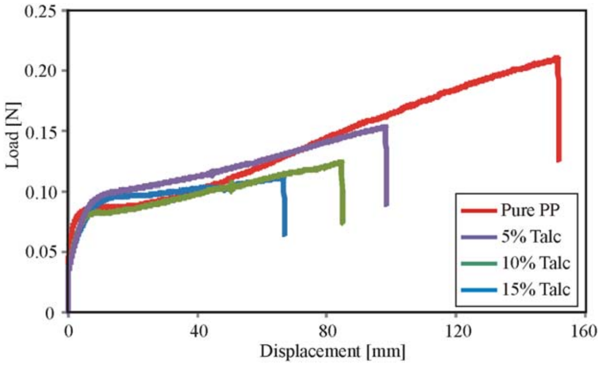

Figure 3. Typical load–displacement curves of pure PP and 5%, 10%, and 15% talc-filled PP samples with gage length of 50 mm. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

63.2% for a specimen with $V = V_0$. For a set of nominally identical samples in tensile testing (i.e., $V = V_0$), the cumulative failure probability is

$$P(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{4}$$

and its pdf is

$$P(\sigma) = \frac{m}{\sigma_0}\left(\frac{\sigma}{\sigma_0}\right)^{m-1}\exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{5}$$

By maximizing its log-likelihood function, the two unknown parameters in the Weibull distribution can be calculated as

$$\sigma_0 = \left\{\frac{1}{n}\sum_{i=1}^{n}\sigma_i^m\right\}^{1/m} \quad \text{and} \quad \frac{1}{m} = \frac{\sum_{i=1}^{n}[\sigma_i^m \ln(\sigma_i)]}{\sum_{i=1}^{n}\sigma_i^m} - \frac{1}{n}\sum_{i=1}^{n}\ln(\sigma_i) \tag{6}$$

where σ_i is strength of the ith sample and n is the number of samples or tests. The strength distribution of a material may be symmetrical and therefore the normal distribution is a potential distribution to fit its strength data.[41]

The pdf of a normal distribution can be represented as

$$P(\sigma) = \frac{1}{\sqrt{2\pi}\alpha}\exp\left[-\frac{(\sigma-\bar{\sigma})^2}{2\alpha^2}\right] \tag{7}$$

where $^-$ and α^2 are the mean and variance, respectively. Their maximum likelihood estimators are

$$\bar{\sigma} - = \frac{1}{n}\sum_{i=1}^{n}\sigma_i \quad \text{and} \quad \alpha^2 = \frac{1}{n}\sum_{i=1}^{n}(\sigma_i - \bar{\sigma})^2 \tag{8}$$

The log–normal distribution is a distribution of a random variable whose logarithm is normally distributed. Log–normal distribution is also used for characterization of molecular weight and fiber properties of polymers and nonwovens.[42–45] Its pdf can be written as

$$P(\sigma) = \frac{1}{\alpha\sigma\sqrt{2\pi}}\exp\left[-\frac{(\ln\sigma - \bar{\sigma})^2}{2\alpha^2}\right] \tag{9}$$

where the mean of a log–normal distribution is $\exp(\bar{\sigma}+\alpha^2/2)$ and the variance is $\exp(2\bar{\sigma}+\alpha^2)[\exp(\alpha^2)-1]$. For maximum likelihood estimators, the same procedure as the normal distribution can be used.

RESULTS AND DISCUSSION

In this study, from each type of talc-filled PP fibers (5, 10, and 15% talc filled and pure PP), 55 samples have been used for the tensile testing with the gage lengths of 12.5, 25, 50, 75, and 100 mm. Typical load-displacement curves of pure PP, 5, 10, and 15% talc-filled PP samples with gage length of 50 mm are given in Figure 3. It is seen that the load carrying capacity and elongation at break values are decreasing by increasing the talc ratio. Moreover, the strain hardening exponent (measure of the nonlinearity of the curve) in plastic region is the highest in pure PP sample. The higher the strain hardening exponent, the greater the increase in flow stress and the greater the tendency for plastic deformation to become uniform. As strain hardening exponent increases, the yield strength and the ductility of the material also increases. Unlike pure PP, talc filled PP samples have a smaller plastic region and the plastic hardening part of the curve is almost constant. As a result, it can be said that addition of talc particles decreases the yield strength and ductility.

In Figure 4, the load–displacement curves of 10 samples from 15% talc-filled PP and pure PP are shown. Load values show a scatter in both cases. The scatter in load is larger in 15% talc-filled PP samples. Due to this scatter, a probabilistic method instead of deterministic method is used for the characterization of the strength of investigated samples.

First of all, discrimination of tensile strength distributions by using the three most versatile distribution functions (i.e., Normal, Log–normal, and Weibull) is presented. In the standard ASTM D3822, it is suggested to use gage a length of larger than 10 mm. For the selection of best fitting distribution functions,

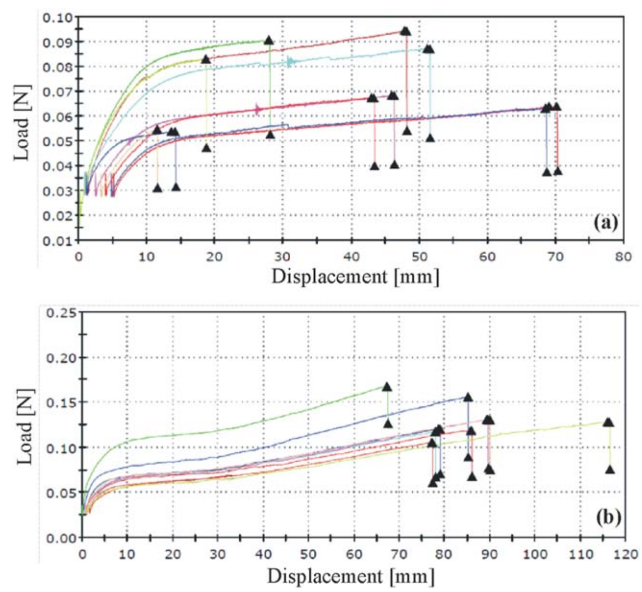

Figure 4. Load–displacement curves of (a) 15% talc-filled PP and (b) pure PP samples with gage length of 50 mm. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table I. $\Delta(\text{AIC})_{normal}$ or $\Delta(\text{AIC})_{log\text{-}normal}$ Values of Tensile Strength of Talc-Filled PP Fibers Containing Different Amount of Talc Ratio

Talc ratio (%)	$\Delta(\text{AIC})_{normal}$	$\Delta(\text{AIC})_{log\text{-}normal}$
15	−8.9	−20.1
10	−1.7	−7.8
5	−0.3	−7.5
0	5.9	−23.45

the strength measurements performed with 50 mm gage length. The AIC has been used for the comparison of fitting performances of three probability functions according to $\Delta(\text{AIC})_{normal}$ and $\Delta(\text{AIC})_{log–normal}$ values which are calculated as follows:

$$\Delta(\text{AIC})_{normal} = \text{AIC}_{Weibull} - \text{AIC}_{normal}$$
$$\Delta(\text{AIC})_{log-normal} = \text{AIC}_{Weibull} - \text{AIC}_{log-normal} \quad (10)$$

when comparing two different distribution functions, AIC states that the distribution which provides a smaller AIC value fits the data better. As a result, according to eq. 10, a positive $\Delta(\text{AIC})_{normal}$ or $\Delta(\text{AIC})_{log–normal}$ value states that the normal or log–normal distribution functions fit the data better respectively. $\Delta(\text{AIC})_{normal}$ or $\Delta(\text{AIC})_{log–normal}$ values related to tensile strength of fibers with different talc ratio are given in Table I.

A negative Δ(AIC) value means that the strength data follows the Weibull behavior. For all talc-filled PP fibers with different talc ratios, the Weibull distribution function fits the data better. For the pure PP fibers, it is seen that the strength data is normal distributed since $\Delta(\text{AIC})_{normal}$ value is positive. As the talc ratio increases from 5 to 15%, the $\Delta(\text{AIC})_{normal}$ and $\Delta(\text{AIC})_{log–normal}$ increases in the negative direction, which means that the distribution of strength data approaches to Weibull behavior more.

Addition of talc particles changes the failure behavior of PP fibers. The regions, where the talc particles are located behave as stress concentration points. In pure PP, failure happens due to the break of secondary bonding between the molecules so the failure occurs in atomic scale. However, in talc-filled PP, failure starts at the stress concentrated regions and therefore the failure mechanism of talc-filled PP fibers occurs at microscopic scale. The position, the size and the shape of the talc particles in the PP fiber determine the failure of the fiber and this is explained by the "weakest-link theory," which is the basic principle of Weibull distribution function. Unlike talc-filled PP fibers, the strength data of pure PP fibers are distributed symmetrically since the failure of the fiber occurs at atomistic scale. For all samples experimented with different gage lengths, the same trend has been observed. Therefore, Weibull distribution function is selected for the strength characterization of investigated fibers in determination of influence of gage length and sample size.

Influence of Gage Length

In Figure 5, the Weibull modulus and the characteristic strength of investigated fibers are shown as a function of gage length. Weibull parameters have been calculated using **Maximum-Likelihood method.[46] For pure PP fibers, the gage length does not have a strong effect on Weibull modulus and characteristic strength. Similar results have also been reported in the study of Schwartz *et al.*,[22] in which it was stated that gage length (10–200 mm) has no influence on strength ultra-high strength polyethylene fibers

In Figure 5(a) it can be seen that as the talc ratio increases up to 5%, an increase in the mean strength of the fibers does not change too much. However, further increase of talc ratio causes a reduction in the tensile strength of PP fibers since the talc particles staying in the fiber behave as stress concentration points. The larger number of talc particles in certain regions causes more stress concentration points and more critical vacancies, which initiates failure of the fibers at lower loading. Therefore an increase in gage length results a larger number of talc particles and a decrease in the characteristic strength of talc-filled PP fibers [see Figure 5(a)]. An increase in gage length results in tensile loading of a larger volume and as the volume increases, the probability of having more critical stress concentration point increases. A more critical stress concentration point then causes a lower strength. This is easily seen on the trend that, the change of tensile strength as a function of gage length also increases as the talc ratio increases. A decrease in tensile strength with an increase of deformed volume/area is defined as "size effect."[47] From three distribution functions, Weibull distribution is the only distribution, which can characterize this effect. This is also another reason why the strength of talc-filled PP fibers follows the Weibull distribution.

According to Figure 5(b), the Weibull modulus increases as the gage length increases for the talc filled PP fibers. Physically, a

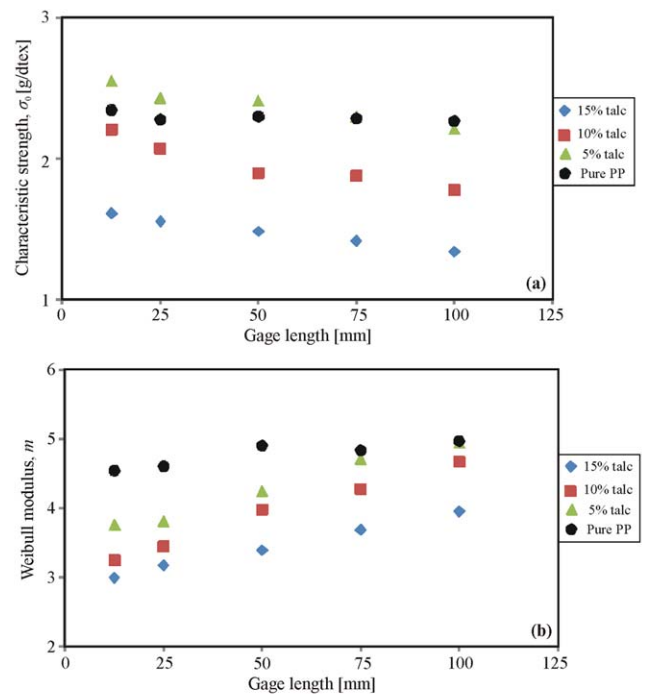

Figure 5. Influence of gage length on (a) Weibull modulus, *m* and (b) characteristic strength, σ_0 of PP fibers filled with different talc ratios. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

larger Weibull modulus means a smaller strength scattering. As the gage length increases, the possibility of having more critical points with similar sizes of talc particles and therefore the strength values of the same type of fibers become closer to each other. That is why strength results of fibers made from 100% PP are independent from gage length of the strength test and show higher Weibull modulus values. Furthermore, as seen in Figure 3, the elastic region of talc filled PP fibers becomes smaller and this shows that the fiber becomes more brittle.

Influence of Sample Size

The strength results given in Figure 3 show that the addition of talc particles caused the fiber to behave in a more brittle nature. Furthermore, if the positions of talc particles in talc filled PP fiber are assumed as points, where stress concentrations occur and failure starts, it is expected that the number of samples for the strength tests plays an important role in determining the tensile strength of the fiber.[23,32]

To investigate the effect of sample size (the number of tests used in tension test) on Weibull parameters of strength of talc-filled PP fibers and determine the minimum required number of samples for a reliable estimation of mechanical properties, 250 tensile tests have been performed with the samples containing 10% talc particles. The Weibull parameters of the 250 experiments, calculated by using the Maximum Likelihood method[46] are, $m = 3.67$ and $\sigma_0 = 1.85$ g/dtex. These Weibull parameters will be taken as global Weibull parameters (m_{gl}, $\sigma_{0,gl}$).

For the investigation of the effect of sample size on Weibull parameters of the tensile strength of talc-filled PP fibers, a computer code was written in MATLAB. Let us assume that there are j different sample groups. Each group contains the number of strength data from $n = 10$ to 200. In each sample group, strength data are randomly selected from the experimental data set and then ranked in an ascending order. Then the Weibull parameters were estimated by using Maximum Likelihood method for each sample group. This procedure was repeated 10,000 times using the MATLAB code. For each sample group, mean, minimum and maximum values of Weibull modulus and characteristic strength of 10,000 simulations were recorded. In Figure 6, the recorded Weibull modulus and characteristic strength values are given as a function of sample size. The range of values that Weibull parameters take for each sample size from 10,000 simulations is shown by error bars.

It is clearly seen that when 20 samples are used for the determination of strength as suggested by ASTM D3822 standard, the range of the Weibull modulus values (2–14) is very large [see Figure 6 (a)]. Let us take a fiber which is loaded by a tensile stress of 1 g/dtex. Using Weibull modulus of 14 instead of 2 decreases the calculated failure probability by almost 1,400 times and using Weibull modulus of 14 instead of 3.67 (true Weibull modulus) decreases the calculated failure probability by almost 600 times. This shows that using a Weibull modulus larger than the true Weibull modulus results in a calculation of lower failure probability. Therefore using 20 samples for finding the average tensile strength and Weibull parameters cause an unreliable design. Furthermore, it is seen that the number of data sets,

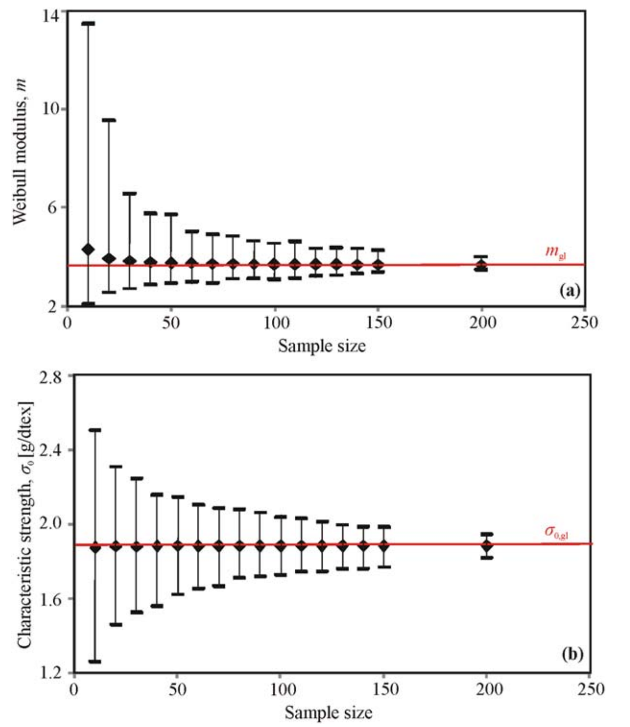

Figure 6. (a) Weibull modulus, m and (b) characteristic strength, σ_0 as a function of sample size of specimens tested with gage length of 50 mm. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

which give Weibull modulus larger than global value is higher for all sample sizes. However, the mean of 10,000 data sets for each sample size does not change noticeably.

In Figure 6(b), the characteristic strength values are given as a function of sample size. The mean of 10,000 simulations is not dependent on the sample size. However, the range of the characteristic strength values decreases as the sample size increases. Characteristic strength is somehow the mean of the strengths of tested specimens and as the sample size increases, the mean values of 10,000 simulations become closer to each other. Since the sample size is large, a very high strength or a very low strength does not affect the mean so much. However, for small sample sizes, this effect becomes more visible.

For each sample size, 90% confidence interval of the Weibull parameters of each of 10,000 data sets were compared with global Weibull parameters as given below,

$$\begin{gathered} m_{i,L} \leq m_{gl} \leq m_{i,U} \\ \sigma_{0,i,L} \leq \sigma_{0,gl} \leq \sigma_{0,i,U} \end{gathered} \tag{11}$$

Here $m_{i,L}$ and $\sigma_{0,i,L}$ lower limits and $m_{i,U}$ and $\sigma_{0,i,U}$ are the upper limits of 90% confidence interval of Weibull parameters of ith data set where i is from 1 to 10,000. If the global Weibull parameter is between these two limits, it can be said that the 90% confidence intervals of the calculated Weibull parameters includes the global Weibull parameters and we are partially in the reliable side. The number of calculated Weibull parameters of 10,000 simulations that satisfy the condition given in eq. 11,

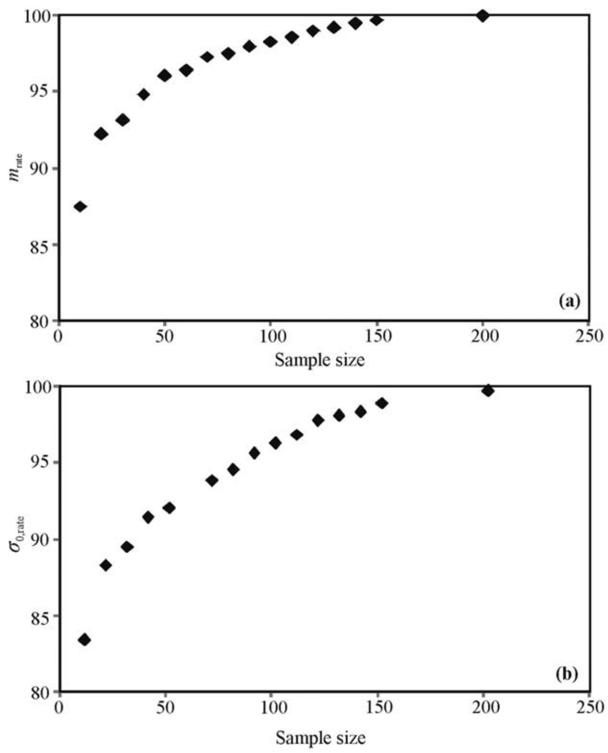

Figure 7. (a) m_{rate} and (b) $\sigma_{0,rate}$ as a function of sample size.

are recorded as m_{rate} and $\sigma_{0,rate}$ and their changes with the sample size are shown in Figure 7.

If 10 samples are used for the determination of the Weibull parameters, 87% of Weibull modulus and 83% of σ_0 values of 10,000 simulations satisfy the condition given in eq. [11]. This means that almost 15% of the calculated Weibull parameters are unreliable. In case the designer does not use 90% confidence interval, the ratio of the calculated Weibull parameters in the unreliable region is much higher than 15%. According to these results, for 95% reliability of calculating of correct Weibull parameters, it is suggested to use at least 50 specimens in tensile tests for the determination of mechanical properties when talc particles are filled to pure PP fibers.

CONCLUSIONS

In the framework of this study, talc-filled PP fibers containing various talc ratios were produced and tensile tests were performed with various gage lengths. By using the experimental results, the reliable determination of strength of talc-filled PP fibers was investigated. According to the results, the following conclusions are made;

- Addition of talc particles into pure PP results in a decrease in strain-hardening exponent as well as the yield strength and ductility of the fiber. Due to the brittle nature of talc-filled PP fibers, a scatter of tensile strength appears.
- Akaike information criterion results showed that the Weibull distribution function is the best function for characterization of talc-filled PP fibers. Moreover, as the talc ratio increases, the strength distributions talc-filled PP fibers approach to Weibull distribution more.
- During experimentation, fiber samples were tested with different gage lengths (12.5, 25, 50, 75, and 100 mm). For pure PP fibers, it was observed that the influence of gage length on Weibull parameters is very small and ignorable. However, for talc-filled PP fibers, as the gage length increases, the characteristic strength decreases and Weibull modulus increases.
- According to ASTM D3822, 20 samples are suggested for the tensile strength determination. However, this is suggested for pure fibers. It was observed in this study that at least 50 samples should be used for a reliable Weibull parameter determination when talc particles are added into the pure PP fiber.
- For different mineral particle-filled PP fibers, the reliability of ASTM D3822 standard should be questioned according to experimental setup parameters and minimum sample size.
- As a future work of this study, samples with different fillers with various filler sizes will be produced and the effects of filler type, filler size, filler size/fiber diameter ratio, uniform distribution of filler within the material and interfacial modification will be examined by using SEM microstructure images.

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REFERENCES

1. Silva, L. P.; Barbosa, E. F. Polypropylene: Synthesis, Applications and Environmental Concerns (Polymer Science and Technology); Nova Science Publishers: New York, **2013**.
2. Fu, S. Y.; Lauke, B.; Mader, E.; Yue, C. Y.; Hu, X. *Comp. Part A* **2000**, *31*, 1117.
3. Etcheverry, M.; Barbosa, S. E. *Materials* **2012**, *5*, 1084.
4. Jambrich, M.; Hodul, P. In Polypropylene—Polymer Science and Technology Series; Springer: Netherlands, **1999**; Vol. *2*, pp 806–812.
5. Karger-Kocsis, J. In Polypropylene Structures, Blends and Composites; Springer: Netherlands, **1995**; Vol. *3*, pp 141–161.
6. Erdem, N.; Erdogan, U. H.; Aksit, A. *J. Text. Sci. Eng.* **2008**, *69*, 15.
7. Naebe, M.; Hurren, C.; Maazouz, A.; Lamnawar, K.; Wang, X. *Fiber Polym.* **2009**, *10*, 662.
8. Srisawat, N.; Nithitanakul, M.; Srikulkit, K. *J. Met. Mat. Min.* **2009**, *19*, 53.
9. Dabrowska, I.; Fambri, L.; Pegoretti, A.; Slouf, M.; Vackova, T.; Kolarik, J. *Exp. Polym. Lett.* **2015**, *9*, 277.
10. Rottstegge, J.; Zhang, X.; Zhou, Y.; Xu, D.; Han, C. C.; Wang, D. *J. Appl. Polym. Sci.* **2007**, *103*, 218.
11. Caldas, V.; Brown, G. B.; Nohr, R. S.; Macdonald, J. G.; Raboin, L. E. *J. Appl. Polym. Sci.* **1997**, *65*, 1759.
12. Takahashi, T.; Kimura, Y. *Text. Res. J.* **2012**, *82*, 789.

13. Broda, J.; Slusarczyk, C.; Fabia, J.; Demsar, A. *Text. Res. J.* **2016**, *86*, 64.
14. Jeong, S. H.; Yeo, S. Y.; Yi, S. C. *J. Mater. Sci.* **2005**, *40*, 5407.
15. Bevitori, A. B.; Da Silva, I. L. A.; Lopes, F. D. P.; Monteiro, S. N. *Rev. Mater.* **2010**, *15*, 117.
16. Inacio, W. P.; Lopes, F. P. D.; Monteiro, S. N. *Rev. Mater.* **2010**, *15*, 124.
17. Joffe, R.; Andersons, J.; Sparnins, E. In Proceedings of the ICCM 17 Edinburgh, Edinburgh International Convention Centre, Edinburgh, UK. IOM Communications, London, **2009**.
18. Sia, C. V.; Nakai, Y.; Shiozawa, D.; Ohtani, H. *Open J. Compos. Mater.* **2014**, *4*, 72.
19. Wagner, H. D.; Phoenix, S. L.; Schwartz, P. A. *J. Compos. Mater.* **1984**, *18*, 312.
20. Amaniampong, G.; Burgoyne, C. *J. Mater. Sci.* **1994**, *29*, 5141.
21. Lim, J.; Zheng, J. Q.; Masters, K.; Chen, W. W. *Int. J. Impact Eng.* **2011**, *38*, 219.
22. Schwartz, P.; Netravali, A.; Sembach, S. *Text. Res. J.* **1986**, *56*, 502.
23. Naito, K. *J. Appl. Polym. Sci.* **2013**, *128*, 1185.
24. Tascan, M.; Nohut, S. *Text. Res. J.* **2016**, DOI: 10.1177/004051751562215.
25. Nohut, S. *Ceram. Int.* **2012**, *38*, 2411.
26. ASTM D3822. Standard Test Method for Tensile Properties of Single Textile Fibers, **2007**.
27. Tascan, M.; Nohut, S. *Text. Res. J.* **2014**, *84*, 2214.
28. Nohut, S.; Lu, C. *Ceram. Int.* **2012**, *38*, 4979.
29. Lu, C.; Danzer, R.; Fischer, F. D. *J. Am. Ceram. Soc.* **2002**, *85*, 1640.
30. Pickering, K. L.; Murray, T. L. *Compos. Part A: Appl. Sci. Manuf.* **1999**, *30*, 1017.
31. Wilson, D. M. *J. Mater. Sci.* **1997**, *32*, 2535.
32. Xia, Z. P.; Yu, J. Y.; Cheng, L. D.; Liu, L. F.; Wang, W. M. *Compos. Part A: Appl. Sci. Manuf.* **2009**, *40*, 54.
33. Berger, M. H.; Jeulin, D. *J. Mater. Sci.* **2003**, *38*, 2913.
34. Barella, A. *Text. Res. J.* **1965**, *35*, 1051.
35. Warburton, C. E. *Text. Res. J.* **1967**, *37*, 703.
36. Li, T. T.; Wang, R.; Lou, C. W.; Lin, J. Y.; Lin, J. H. *Text. Res. J.* **2014**, *84*, 1903.
37. Anandjiwala, R. D.; Goswami, B. C. *Text. Res. J.* **1993**, *63*, 392.
38. Wang, F.; Shao, J. *Polymers* **2014**, *6*, 3005.
39. Lu, C.; Danzer, R.; Fischer, F. D. *Phys. Rev. E* **2002**, *65*, 067102.
40. Dirikolu, M. H.; Aktas, A.; Birgoren, B. *Turkish J. Eng. Env. Sci.* **2002**, *26*, 45.
41. Reutov, Y. I.; Reutov, A. I.; Sidorenko, A. A. In Proceedings of the Fifth Russian-Korean International Symposium on Science and Technology, Tomsk, Russia, **2001**; pp 217–219.
42. Conti, W.; Gigli, I. *J. Polym. Sci. Part A: Polym. Chem.* **1996**, *4*, 1093.
43. Singha, K.; Maity, S.; Singha, M.; Paul, P.; Gon, D. P. *Int. J. Text. Sci.* **2012**, *1*, 7.
44. Ellison, C. J.; Phatak, A.; Giles, D. W.; Macosko, C. W.; Bates, F. S. *Polymer* **2007**, *48*, 3306.
45. Rawal, A. *J. Text. I.* **2010**, *10*, 350.
46. Ambrozic, M.; Gorjan, I. *J. Mater. Sci.* **2011**, *46*, 1862.
47. Nohut, S.; Schneider, G. A. *J. Eur. Ceram. Soc.* **2009**, *29*, 1013.