Study on the effect of hybrid fiber length on tensile strength of sealing composites

Li Yuxian¹, Liu Meihong¹, Sun Junfeng¹, Wang Juan¹ and Tian Shuo²

Abstract
The present paper aims at investigating the relationship between fiber length of hybrid fibers and the tensile strength of sealing composite materials. First, three kinds of fibers: cellulose fiber, aramid pulp fiber, and mineral wool fiber were sieved and their weight-average length was measured. Second, a uniform design method of U8 (4³) was adopted to prepare sealing composites by the beater-addition process, and the properties of the tensile strength of the composite were examined. In the end, the relation model was concluded and verified using multi-linear regression analysis and was further analyzed using micro mechanic theory and interfacial bonding mechanism. The results show that the regression equation can be used to estimate the tensile strength of composites with different hybrid fiber lengths. The tensile strength increased corresponding to the increase of the length of the cellulose fiber but decreased with the increase of the length of aramid pulp and mineral wool fiber. Particularly the fiber length of aramid pulp fiber had the most significant effect on tensile properties. The cases were decided by the comprehensive effects of fibers dispersion, interfacial bonding, and micromechanics.

Keywords
Fiber length, tensile strength, hybrid fiber, sealing composite, regression analysis, interfacial bonding, micromechanics

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Introduction
Fiber reinforced composite (FRC) is a much researched material.¹ Sealing composite is mainly used for the sealing connection of processing equipment, pressure vessels and pipe networks within industries of chemical, petroleum, electric power and medicine. In order to promote public health and sustainability, traditional asbestos fibers have been banned at in many countries across the globe. Sealing composite is usually prepared by non-asbestos fibers, fillers, latex, and chemical assistants through a specialized process.²³ After decades of development, non-asbestos sealing composites have been ever widely applied across industries, but there is still a gap in performance compared to asbestos products. As a sealing material, to reduce leakage, the composite is required to have high levels of rigidity and resistance to pressure. This performance can be measured indirectly using tensile strength. Tensile strength can be used as one of important evaluation indicators for properties of sealing composites.⁴⁵

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Fibers play an important reinforcing role in sealing composite. The properties of composites are closely related to the types, properties, ratio, and morphology of their fibers. In order to effectively replace asbestos fibers, researchers have been undertaken seeking the optimal formula with best performance, a lot of work has been done on the selection of reinforcing fibers and surface treatment. To date, no single substitute fiber which can match asbestos fiber has been found. The formulation design of non-asbestos composite sealing material is significantly more complicated than that of traditional asbestos products. At present, considering performance and cost, the hybrid of organic fiber, plant fiber and inorganic fiber is best placed to replace asbestos.

For a given fiber, the morphology is one of the important factors affecting the mechanical properties of a composite material. Some academics have studied the mechanical properties of short fiber reinforced composites with different types of fibers. Vairavan et al. investigated the effect of fiber length and fiber content of basalt fiber on mechanical properties of fabricated composites. Sreenivasulu studied the variation of tensile strength with varied fiber length of short bamboo fiber reinforced composites. Sun et al. obtained the formula for predicting longitudinal elastic modulus of short-fiber-reinforced composite viewed according to length influence factor and the mixing rule. The results showed that the micro-structure parameters, including fiber aspect ratio and volume fraction have a great influence on longitudinal elastic modulus of the composite. Yao et al. discussed the effect of aramid fiber length on paper structure and mechanical properties, and the results showed that the density of paper decreased with the increasing of Fiber length. In the range of 1–4 mm, tensile strength, percentage of elongation, and burst strength increased; however, above 4 mm, there was no significant change. Bisaria et al. prepared composite with jute fiber in various lengths of 5, 10, 15, and 20 mm into epoxy matrix. The results showed that the tensile and flexural properties were found to be greatest for the composite with 15 mm length of fiber. Wu built a tensile strength prediction model for flax fiber reinforced epoxy resin composite. The results showed that the critical length of the fiber, tensile strength and fiber length effect factor were different because of the different shear strength for composites of different fiber contents. Wang and Wang proposed a new model for predicting discontinuous natural fibers reinforced biodegradable composite. The model contains not only the probability effect for distributing features of the fibers strength, length, fibers tropism angle and diameter on the predicted strength, but also the effect of the restricted connection among the shear strength of the composite, the critical length of the fibers and the terminal tensile strength of the fibers in the composite based on strength. Liu et al. studied the influence of the residual length of the glass fiber on the mechanical properties of glass fiber reinforced thermoplastic composites. The results show that the smaller the shear strength of the screw, the longer the fiber length retains in the product. The bonding interface between the fiber and the matrix increases corresponding to the increase of the glass fiber length. Larger interfacial adhesion between the glass fiber and the substrate must be overcome during withdrawal. Bian et al. studied the effects of the pretreatment method and length of aramid short fiber on the properties of the aramid short fiber reinforced composites. The results showed that the composites with 3 mm aramid short fiber were more robust than those with 1 and 6 mm aramid short fiber, when the amount of addition were equal.

From the above studies, the influence of fiber length on the mechanical properties of composites can be understood as being highly complex. At present, most of the studies only discuss single fiber reinforced composites, mainly focusing on unidirectional short fiber reinforced composites. As to non-asbestos sealing materials with hybrid and random distribution of fibers, there remains a lack of comprehensive research. The selection of fiber parameters in production is still dependent on operational experience.

Considering the above thesis, this paper chose hybrid fibers of aramid pulp, cellulose fibers, and mineral wool to prepare sealing composite. Through experimental design, keeping the formula and process conditions unchanged, the influence of different fiber length on the tensile strength of the composite was studied. Employing regression analysis, the relationship between tensile strength and fiber length was obtained and verified. At the same time, the relationship was analyzed from the perspective of micro-mechanics and interface theory. The studies provide useful guidance for the selection of fiber length and predicting composite properties in production, and providing an important experimental basis for the continuous improvement of micro reinforcement mechanism of short fiber reinforced composites.

**Experimentation**

**Materials**

Materials include three kinds of fiber bundles: aramid pulp, cellulosic fiber, and mineral wool fiber; fillers: calcined kaolin, French chalk, and mica; Latex binder: Nitrile latex and styrene-butadiene latex; vulcanization accelerator TMTD, vulcanization active agent ZnO, and vulcanizing agent S; and chemical additives including dispersants, flocculants, and defoamer. Among these, aramid pulp: average diameter is 12 μm, average length range is approximately 0.5–1.5 mm, density is 1.44 g/cm³; cellulose fiber: diameter range is 5–25 μm, single fiber length is approximately 1.5–5 mm; mineral cotton 4012 MF: diameter range is 3–15 μm, average length range is 1.2–3 mm, density is 2.75 g/cm³. The above were all supplied by Chengdu Tianfu Gasket Technology Co. Ltd. (Chengdu, China).
Fiber length measurement

Fiber sieving. The fiber bundles need to be sieved to obtain different lengths. In this case, BAUER McNett Fiber Classifier ZT10-00 was employed. The relatively dilute fiber suspension continuously flowed through several screens with different mesh sizes, different lengths of fiber were retained on each sieve, and the length of the retained fiber complimented the mesh aperture of the sieve plate. The screens are medium length fiber screens, and the meshes are: 14, 28, 48, and 100 mesh. A certain weight of fiber suspension of cellulose fiber, aramid pulp, and mineral wool fiber was taken for sieving respectively, the frequency of the agitator of the Bauer sifter was adjusted to 20 Hz, and the sieving time was set at 45 min. The fibers on each sieve were collected to obtain fibers of different lengths. The sieved fibers were dried for later use.

Fiber length measurement. The number average fiber length is also described as the arithmetic average fiber length, which is the arithmetic average of the total length of the measured fiber divided by the total number of fibers:

\[ L_n = \frac{\sum n_i L_i}{\sum n_i} \]  

Where \( L_n \) is number average fiber length. \( n_i \) is the number of fibers of each component been measured. \( L_i \) is the average length of the fibers measured.

Because the length of \( L_n \) is affected by the number of fine fibers, the characteristics of the fiber cannot be accurately expressed by \( L_n \). \( L_n \) does not accurately reflect the length of most fibers, nor can it truly reflect the level of damage fiber.

According to GB/T10336-1989 of China, mass average fiber length, is known as length weighted average length (and also known as weight-average fiber length). The specific method is as follows: firstly, calculate the product of measured average fiber length for each length group and for the corresponding component mass, followed by the sum of product divided by the total mass. Expressed by the following formula:

\[ L_w = \frac{\sum m_i L_i}{\sum m_i} \]  

where \( L_w \) is weight-average fiber length. \( m_i \) is the measured mass of each component fiber? \( L_i \) is the average length of each component fiber measured. \( L_w \) is closer to the real situation of fiber destruction than \( L_n \). So this article adopts weight-average length. The weight-average length of the above sieved fibers measured by HiRes analyzer is as follows in Table 1.

Uniform experimental design

In order to study the effects of different lengths of cellulose fibers, aramid pulp, and mineral wool fibers on the properties of composite materials, a uniform experimental design was performed for different lengths of these three fibers. Components, formula, and process conditions remain unchanged, and only the length of the three fibers is changed. According to the fiber length obtained from the previous screening of fibers, the values of independent variables are shown in Table 2.

According to the research of this paper and the actual needs of the experiment, it was decided to adopt uniform experimental scheme of U8 (4³), the scheme of uniform scale is D = 0.2000, the smaller the D value, the more uniformly the design points will be. It also shows that the designed experiment is more representative. However, due to the small number of factors selected in this experiment, it is challenging to meet the use requirements of U8 (4³), the quasi-level method is adopted to fit the test level, and several levels are fitted to meet the use requirements of U8 (4³). The experimental design scheme after fitting is shown in Table 3.

Specimen preparation

The composite materials were prepared using a beater-addition process. The screened cellulose fiber, aramid

Table 1. Weight-averaged length of different mesh number of fibers after screening.

| Mesh number (Mesh) | Cellulose fiber (μm) | Aramid pulp fiber (μm) | Mineral wool fiber (μm) |
|-------------------|----------------------|------------------------|------------------------|
| 14                | 2650                 | 1222                   | 2420                   |
| 28                | 2234                 | 990                    | 2010                   |
| 48                | 1930                 | 895                    | 1915                   |
| 100               | 1212                 | 560                    | 1210                   |

Table 2. The value range of independent variable.

| Independent variable | Code | Value (μm) |
|----------------------|------|------------|
| Weight-average length of cellulose fibers | X₁ | 2650, 2234, 1930, 1212 |
| Weight-average length of aramid pulp fibers | X₂ | 1222, 990, 895, 560 |
| Weight-average length of mineral wool fibers | X₃ | 2420, 2010, 1915, 1210 |

Table 3. The designed experiment project of U₈ (4³).

| Experiment number | Fiber length (μm) | X₁ | X₂ | X₃ |
|-------------------|-------------------|----|----|----|
| 1                 | (1)2650           | (2)990 | (4)1212 |
| 2                 | (1)2650           | (4)560 | (3)1915 |
| 3                 | (2)2234           | (2)990 | (2)2010 |
| 4                 | (3)2234           | (4)560 | (1)2420 |
| 5                 | (3)1930           | (1)1222 | (4)1210 |
| 6                 | (3)1930           | (3)895 | (3)1915 |
| 7                 | (4)1212           | (1)1222 | (2)2210 |
| 8                 | (4)1212           | (3)895 | (1)2420 |
pulp, and mineral wool were selected according to the specified uniform experimental design, all formulations, and process conditions remain unchanged. The selected fiber was loosening in water employing fiber fluffer PL28-00 (Xiayang Taist Test Equipment Co., Ltd. Xianyang, China). Adding the defibering fiber, filler, latex, vulcanization agent, and chemical auxiliary agent into the aqua pulper PL12-2 (Xianyang Taist Test Equipment Co., Ltd. Xianyang, China) in their order. The slurry suspension was prepared by mixing and stirring. After the suspension was treated with defoaming, flocculation, and water filtration, wet composite was obtained. Finally, the samples were prepared through drying and vulcanization.

**Tensile strength testing**

The tensile strength of sealing composite was measured according to Chinese GBT20671.7 and ASTM F152 using a universal testing machine UTM6000 (Shenzhen suns technology Co., Ltd. Shenzhen, China).

**Scanning electron microscopy**

The samples were pretreated by metal spraying. Then morphology of surfaces were studied under a tungsten filament scanning electron microscopy (SEM) instrument VEGA-3SBH (Tescan, Czech Republic) at an accelerating voltage of 20 kV.

**Fourier transform infrared spectroscopy**

Fourier transform infrared spectroscopy (FTIR) was characterized employing Fourier Transform Infrared Spectrometer (TENSOR27, Bruker, Germany). By means of potassium bromide pellet technique. Datas are recorded in the range from 4000 to 500 cm\(^{-1}\).

**Experiment results and discussion**

The results of the above tests are shown in Table 4.

From the table it can be seen that the tensile strength of different composites varies from 7.86 to 14.98 MPa, indicating that the fiber length has a significant influence on the tensile strength of the composite. It can be seen from the table that the maximum value of tensile strength is 14.98 MPa shown in group 2. When the weight-average length of cellulose fiber, aramid pulp fiber and mineral wool fiber in this component is 2650, 560, and 1915 μm respectively. The lowest tensile strength was shown in group 7, the minimum was 7.86 MPa, when the weight-average length of cellulose fiber, aramid pulp fiber, and mineral wool fiber was 1210, 1222, and 2010 μm respectively.

| Experiment number | Fiber length (μm) | Tensile strength (MPa) |
|-------------------|------------------|------------------------|
| 1                 | (1)2650 (2)990 (4)1210 | 12.88                  |
| 2                 | (1)2650 (4)560 (3)1915 | 14.98                  |
| 3                 | (2)2234 (2)990 (2)2010 | 11.032                 |
| 4                 | (2)2234 (4)560 (1)2420 | 12.97                  |
| 5                 | (3)1930 (1)1222 (4)1210 | 10.3005                |
| 6                 | (3)1930 (3)895 (3)1915 | 9.89                   |
| 7                 | (4)1212 (1)1222 (2)2010 | 7.86                   |
| 8                 | (4)1212 (3)895 (1)2420 | 8.25                   |

**Regressive analysis**

The regression equation of tensile strength and fiber length can be obtained by fitting the above test results with SPSS software and multiple linear regression analysis. There were eight sets of data in the experiment, group 8 was reserved for inspection, and regression analysis of another seven groups of data results in the regression equation as follows:

\[
Y = 13.765 + 0.002X_1 - 0.005X_2 - 0.001X_3
\]  (3)

In general, if a regression equation is used to predict performance and optimal formulation, the equation itself must be a significant regression equation. The correlation factor \(R = 0.974\) between independent variables and dependent variables in the above formula, the judgment factor of fitted linear regression \(R^2 = 0.949\), the adjusted \(R^2 = 0.898\), and the significance level \(P = 0.019\) can be obtained through the fitting analysis of experimental data using SPSS software. The above regression model and related parameters of test data showed that: the adjusted \(R^2\) value was close to 0.9, it indicated that the data fitting effect was ideal, and the value of significance level of \(P\) is less than 0.05. This indicated that the whole explaining variation of the regression model can meet the significant level, so it can determine that the regression equation was effective, and holds relevant application value.

Through the obtained regression model, the properties of the composites prepared with different lengths of the three fibers can be predicted. For example, the predicted value of the eighth group of samples is 9.29 MPa, the relative error is 11.19% compared with the experimental data. The difference between the predicted value and the experimental one is not significant. This also shows from another aspect, the regression model of the relationship between fiber length and tensile strength obtained by SPSS software is correct. This regression equation can be used to estimate the tensile strength of composite materials prepared by hybrid fiber of different lengths.

When the length of other fibers remains unchanged, relationships between the length of one fiber in the formula and the composite performance can be investigated according to the regression model. For example, when...
investigate the relationship between the tensile strength of the composite and the length of mineral wool fiber, it can keep the length of the other fibers constant firstly, such as assign the fiber length of cellulose fiber $X_1 = 1210$, the fiber length of aramid pulp $X_2 = 560$, then the relationship between the tensile strength of the sealing composite and the weight-average length of mineral wool fiber can be obtained as shown in Equation 4:

$$Y_3 = 13.385 - 0.001X_3$$  \hspace{1cm} (4)

And so on, when the other conditions keep unchanged the effect of each fiber’s weight-average length on the tensile strength of the composite can be expressed. For example, the length of every fiber was assigned as $X_1 = 2234$, $X_2 = 990$, and $X_1 = 2010$ respectively, the effects of length on tensile strength for each fiber can be shown as Figure 1.

It can be seen from the above figure that the tensile strength of the composite is obviously affected by the fiber length, and the effects vary according to fiber type. With the increase of the length of cellulose fiber the tensile strength of composites gradually increases. With the increase of the length of aramid pulp and mineral wool fiber, the tensile strength of composites decreases gradually, and the length of aramid pulp fiber has the most significant effect on the tensile properties.

**Micro reinforcing mechanism analysis**

According to the stress transfer theory, the minimum fiber length of the allowable fiber strength can be achieved for the specified diameter of the fiber and the specific fiber-matrix interface environment, also known as critical length $l_c$.

$$l_c = \frac{\sigma_{fu}}{2T_i}d_f$$  \hspace{1cm} (5)

where $\sigma_{fu}$ is tensile strength of fibers. $l_c$ is maximum load transfer length, also known as “critical fiber length.” $d_f$ is fiber diameter. $T_i$ is shear strength between the fiber and the interface.

The composites material in this paper are short fiber reinforced composites and mainly rely on fiber as reinforcement materials, obviously satisfied $l_f > l_c$.

When $l_f > l_c$, According to the rule of mixture, the tensile strength of the composite material is calculated as follow:

$$\sigma_{cu} = \sigma_{fu} \left(1 - \frac{l_f}{2l_c} \right)\nu_f + \sigma_{mu} \left(1 - \nu_f \right)$$  \hspace{1cm} (6)

where $\sigma_{cu}$ is tensile strength of composite materials. $l_f$ is fiber length. $\nu_f$ is percentage of fiber by volume. $\sigma_{mu}$ is tensile strength of matrix.

Substitute the above equation (5) into equation (6) to obtain the equation as follow:

$$\sigma_{cu} = \sigma_{fu} \left(1 - \frac{\sigma_{fu}d_f}{4T_i l_c} \right)\nu_f + \sigma_{mu} \left(1 - \nu_f \right)$$  \hspace{1cm} (7)

It can be observed that the load transfer needs a good interface. Under the premise of the formula determination, the strength of the composite depends on the interfacial bonding strength and fiber length. The larger the product of $T_i l_f$, the greater the tensile strength of the composite material. As shown in Figure 2(a)–(c), the microscopic morphology of the three types of fibers differs greatly. So for all three fibers, the interfacial shear exhibits different variations with increasing length, leading to different effects on the tensile strength of the composites. As shown in Figure 2(d), the interfacial bonding of composites is determined by the physico-chemical interactions among the fibers, fillers, and the latex. The SEM fracture morphology of composites from the previous second and seventh groups are shown in Figure 2(e) and (f) respectively, we can see that there are a lot of materials adhere to the drawn fibers from the fracture surfaces in Figure 2(e), while the drawn fibers of Figure 2(f) are relatively smooth, which indicates that the interface bonding of the second group of samples is better.

For aramid pulp and mineral wool fiber, with decreasing of fiber length, the crowding factor decreases and leads to better dispersion. At the same time, the specific surface area of the fiber and the number of charges, chemical bonds and chemical groups on the surface of the fiber are all increased, and the adsorption capacity to the filler and latex is enhanced, which greatly improves the interface adhesion of the sealing composite. The increase of $T_i$ exceeds the decrease of $l_f$, and the overall value of $T_i l_f$ increases, so the tensile strength of the composite is also increased.
Aramid pulp fiber is an ideal substitute for asbestos owing to its excellent dispersion and mechanical properties. It is processed from aramid fiber by surface fibrillation. As shown in Figure 2(a), the surface of aramid pulp fiber is feathery with a larger surface area, which has a better capability of adsorption with latex, fillers, and chemical additives. On the one hand, as the fiber length decreases, the unique microscopic surface structure of the aramid pulp fibers resulted in the dramatically increase in interfacial bonding, and on the other hand, the tensile strength of the aramid fibers is significantly higher than that of the other two fibers, which results in the most significant reinforcement in composite materials. Therefore, the length variation of aramid pulp fiber has the most significant impact on composite materials.

For cellulose fiber, although the increase of fiber length will affect the dispersion, hydroxyl groups can be dissociated from cellulose fibers in water, and hydrogen bonds can be easily formed between water and hydroxyl groups. The longer the cellulose fiber was, the larger the area of hydrogen bond on the fiber and the better the stress distribution was. The stronger the hydrogen bond network constructed by the fiber was, the stronger the interface bonding force was. The effect on this bonding force was more significant than the effect on dispersion, so within limits, with the increase of length of cellulose fiber, both $T_r$ and $I_f$ increased, the strength of the composite material increased. FTIR of composites from the previous second and seventh groups are shown in Figure 3(a) and (b) respectively. The absorption peaks for composites were similar with distinct and broad $\sim$OH absorption peaks around 3400–3500 cm$^{-1}$, which can be attributed to the intermolecular H-bonds between the polar hydroxyl and cellulose fiber. The effect of hydrogen bonds would
change the wavenumber of −OH, the stronger H-bonds with shift to the lower wavenumbers. The composite’s spectrograms show the −OH stretch peak at 3440 cm\(^{-1}\) (a: composite of second group), 3448 cm\(^{-1}\) (b: composite of seventh group). It illustrates the effect of hydrogen bonds become weaker from a to b. This is also agreed with the tensile strength properties.

Conclusion
Sealing composite was prepared by hybrid fibers of cellulose fiber, aramid pulp, and mineral wool fiber with different lengths and through beater-addition process. The tensile strength of the composites was examined.

A model for the relationship between hybrid fiber length and tensile strength of composites was obtained by multiple linear regression analysis. The model was proved to meet the significance level and has certain application value. The predicted values of the tensile strength properties of the composite material obtained by the regression equation did not diverge significantly from the experimental values, which indicated that the obtained regression equation can be used to estimate the tensile strength of the composite prepared from several fibers of different lengths.

The regression equation can also characterize the relationship between one of the fiber lengths and the tensile strength of the composite, when other conditions are held constant. The tensile strength of sealing composite materials increased with the decrease of the length of aramid pulp and mineral wool fiber, but increased with the increase of the length of cellulose fiber, and the effect of length of aramid fiber was notable. The effect on the reinforcement of fiber length to composites varied with the fiber type. This was related to the properties of the fibers themselves, their dispersion in wet forming, interfacial bonding, and the micro mechanics of the composites.

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