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Cellulose Microfibers from Salacca Midrib Fiber Isolated by the Mechanical Treatment

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Isolasi Selulosa Mikrofiber dari Serat Pelepah Salak dengan Perlakuan Mekanik

Abstrak

Serat pelepah salak termasuk limbah alam yang melimpah di daerah Turi, Kabupaten Sleman, Daerah Istimewa Yogyakarta. Selulosa mikrofiber dari pelepah salak berhasil diisolasi dengan metode mekanis dan memiliki karakteristik fisik yang baik. Serat selulosa dengan ukuran mikro dapat memperkuat ikatan antara matriks dan serat karena area kontak yang luas. Metode untuk mengisolasi selulosa mikrofiber yaitu dengan pengadukan mekanis pada kecepatan putaran 5.000; 10.000; dan 15.000 rpm. Perlakuan pengadukan mekanis bertujuan untuk melakukan fibrilasi dan mengurangi ukuran serat karena efek putarannya yang tinggi. Karakterisasi dilakukan dengan XRD, FTIR, dan SEM. Hasil uji XRD menunjukkan bahwa pengadukan mekanis tidak merusak indeks kristalinitas selulosa mikrofiber. Indeks kristalinitas raw material yaitu 64,3% naik menjadi 79,1% untuk indeks kristalinitas selulosa mikrofiber. Identifikasi gugus fungsi menggunakan FTIR tidak menunjukkan perubahan senyawa selulosa akibat dari perlakuan mekanis. Pengamatan SEM menunjukkan bahwa selulosa mikrofiber yang diisolasi dari serat pelepah salak berukuran diameter 5-10 µm dengan panjang serat 100-300 µm. Selulosa mikrofiber potensial digunakan sebagai penguat pada mikro komposit dan diekstrak menjadi material nanoselulosa.

Kata kunci: serat pelepah salak, selulosa mikrofiber, perlakuan mekanis

Abstract

Salacca midrib fibers are abundant natural waste in Turi, Sleman Regency, Daerah Istimewa Yogyakarta. Cellulose Microfibers from the salacca midrib fiber have been isolated by mechanical treatment and successfully have good physical characteristics. Cellulose fibers with micro sizes can strengthen the bond effect between the matrix and the fiber due to the vast contact area. The method for isolated cellulose microfibers by mechanical treatment with the rotation speed of 5,000; 10,000; and 15,000 rpm. Mechanical stirring treatment aims to undergo fibrillation and reduces fiber dimensions because of their high rotation. The characterization by XRD, FTIR, and SEM. The XRD results showed that the mechanical stirring treatment did not damage the crystallinity index of cellulose microfibers. The crystallinity index of the raw material is 64.3%, increased to 79.1% for the microfiber cellulose crystallinity index. Identification of functional groups using FTIR did not show any changes in cellulose compounds resulting from mechanical treatment. Morphological observation of fibers by SEM shows that the diameter cellulose microfibers size obtained from salacca midrib fiber ranges from 5-10 µm with 100-300 µm in length. Cellulose microfibers have the potential to be used as reinforcement in micro composites and extracted into nanocellulose materials.

Keywords: salacca midrib fiber, cellulose microfibers, mechanical treatment

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Introduction

The natural fiber is one of Indonesia's abundant natural resources. Indonesia is located in a tropical area with high rainfall so that many types of plants could live and thrive. *Salacca zalacca* is a palm tree species that is one of the abundant natural potentials and has not been widely utilized. The part of the *Salacca zalacca* tree that could potentially be utilized is the midrib. Based on interviews with the local farming community in the Turi area, Sleman Regency, the midrib should be trimmed once a year as much as 6-8 stems (in 1 tree clump) to make it easier to harvest the snake fruit later. The midrib of *Salacca zalacca* trees have been trimmed into waste and only been utilized as compost by being buried in the ground to decompose. Natural fiber components include cellulose, hemicellulose, lignin, pectin, wax, and water-soluble substances (Li *et al*., 2007). In particular, cellulose is the most abundant source of renewable polymers available today. Therefore, it is considered an almost inexhaustible source of raw materials for the increasing demand for environmentally friendly and biocompatible products (Brinchi *et al*., 2013).

Many studies have been carried out to explore the potentials of biodegradable materials derived from nature to be applied as reinforcement in composite material, especially natural fibers. The natural fibers utilized for structural applications were researched and developed from micro to nanometer scale. The midrib fiber could be developed as reinforcement in composites material on the micro and nanometer-sized scale. Cellulose microfibers have been widely developed by researchers for their various method of isolation. One of various isolation methods for the production of cellulose microfibers by mechanical treatment such as by modified kitchen blender with a rotation speed of 21,000 rpm (Sukmawan *et al*, 2019; Saputri *et al*, 2018). Mechanical fibrillation utilizing a modified kitchen blender showed the results of fiber diameter size in the range of 20 nm to 20 µm. A high-speed blender operated at a stirring speed of 37,000 rpm has succeeded in producing nanofiber sized with a uniform diameter of 15-20 nm (Uetani and Yano, 2011).

Cellulose microfiber (CMF) successfully isolated from various types of plants such as banana fiber (Ibrahim *et al*., 2010; Lismeri *et al*., 2017), rubberwood (Lamaming et al., 2016), water hyacinth (Syafri *et al*., 2019), pomelo peel (Liu *et al*., 2018), soybean hulls (Ferrer *et al*., 2016), oil palm empty fruit bunch (Isroi and Cifriadi, 2018), Agave cantala (Rochardjo and Yudhanto., 2019), and cotton fiber (Martínez-Sanz *et al*., 2017). Each natural fiber has different characteristics, thus it is necessary to optimize the parameters to obtain cellulose microfibers with good characteristics for reinforcement. In this study, cellulose microfibers were isolated utilizing mechanical treatment as a candidate for reinforcement in the polymer matrix micro composite material. For further action, microfibers cellulose could be extracted into nanocellulose material. Mechanical treatment was utilized to accelerate the fiber fibrillation process due to its high-speed rotation effect. In addition, the variations of rotation speed and time are discussed for their effect on the crystallinity index of microcellulose fibers.

Materials and Methods

Materials

The raw material of salacca midrib fibers (SMF) obtained from the *Salacca zalacca* (Gaert.) Voss) plant in Turi, Sleman Regency. The salacca midrib fibers are cut in the range of 40-50 cm from the base. Sodium hydroxide (≥ 98% purity), pellets (anhydrous), was purchased from Sigma and Aldrich Chemical Co., Hydrogen peroxide (3% purity), and distilled water were supplied by CV. Wahana Hilab Indonesia, Yogyakarta. All chemicals and reagents were of analytical grade.

Isolation Method

The purification process of cellulose from salacca midrib fibers was isolated by following the previous study procedure of Yudha *et al* (2019). Chemical composition refers to SNI 0492:2008 was determined two times by chlorite acid modification method and lignin for the raw fiber and treated fiber. Dried SMF was cut about ±1 cm before being utilized in this research. Chemical treatment was carried out in two stages, alkalization and bleaching. In alkaline treatment, the fiber was immersed and stirred in NaOH solution with 2% wt. concentration at a temperature of 70˚C for one hour. It was then

proceeded with bleaching treatment immersed in hydrogen peroxide solution with 3% (v/v) concentration at a temperature of 60˚C, pH 10, for one hour. After each stage of treatment, the fibers were neutralized with 2000 mL of distilled water for every 20 g of fibers. Next step, then dried in the oven at a temperature of 70˚C for one hour.

Mechanical treatment with a mechanical stirrer aims to reduce the size of cellulose to micrometer size. Purified cellulose fibers from the previous chemical treatment were dried and cut about ±5 mm. The ratio of fibers (gram) to distilled water (mL) is 1:200. **Figure 1** shows the mechanical stirrer instrument utilized for this research. The mechanical stirrer was operated with a final rotation of 450,000 times with three-time variations at different speeds and processing times (**Table 1**). The variation of rotation speed and time were studied for their effect on the crystallinity index. After the mechanical stirring process, the sample was neutralized with distilled water until the pH was neutral.

Characterization

SEM model JEOL-JSM 6410LA observed the surface and morphology of raw fiber, purified fiber, and cellulose microfiber. The SEM was operated utilizing an accelerating voltage of 3-15 kV with the sample before being coated with a thin layer of Au. Fourier transform infrared (FTIR) aims to analyze the functional group of samples after and before the treatment, the FTIR model is Shimadzu operated in the range of 500-4000 cm^{-1}. XRD instrument identified the structure of the fibers. XRD model is Rigaku which operated at the CuKα radiation wavelength (λ=1.5418 Å) and the 2θ range was 5˚-40˚. Calculation of crystallinity index using the Segal's equation (1) (Maheswari, *et al*, 2012):

$$CrI = \frac{(I_{002} - I_{amorph})}{I_{002}} \times 100\% \quad \text{..... (1)}$$

where I_{002} is the maximum peak intensity at 2θ = 22˚, represents the crystalline cellulose. The minimum peak intensity at 2θ = 18˚ indicated for the amorphous part (Pacaphol and Aht-Ong, 2017).

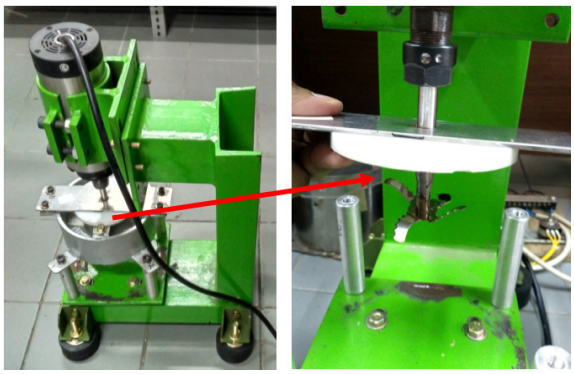

Figure 1. Mechanical stirrer instrument

Table 1. Parameter process for mechanical stirrer

Sample designation	Speed (rpm)	Time (minutes)	Temp. (°C)
M15000	15,000	30	65
M10000	10,000	45	55
M5000	5,000	90	45

Result and Discussion

Purification of Cellulose

The first stage in this research is the purification process of cellulose on the salacca midrib fibers. Purification of cellulose was operated by chemical treatments called alkaline and bleaching. NaOH solution was utilized in the alkaline treatment to remove lignin (Chaker *et al*., 2013), pectin, and waxy substance. Higher cellulose content is better utilized for reinforcing resins because of the cellulose fiber's stiffness (Mwaikambo *et al*., 2002). NaOH has been utilized for alkaline treatment of the fibers with a concentration of 2 wt% at a temperature of ±70°C for 60 minutes. The reaction of cellulose with NaOH is explained in equation (2) (Mwaikambo *et al*., 2002).

$$Cell - OH + NaOH \rightarrow Cell - O^{-}Na^{+} + H_2O + (\text{surface impurities}) \quad \text{.....(2)}$$

The second step of purification treatment is bleaching. The bleaching treatment aims to remove hemicellulose and lignin residues that are still present in the fibers. The bleaching process referred to the previous research by Yudhanto *et al*. (2018), which used 3% H_2O_2 at 60°C and pH 10 for 60 minutes. The ratio of fiber (grams) and H_2O_2 solution (mL) is 1:50. **Figure 2** shows a different visualization of the fiber

Figure 2. Photographs of a) raw fiber, b) alkalized fiber and c) bleached fiber

Table 2. Chemical composition of each cellulose purification

Sample	α-cellulose (%)	Hemi-cellulose (%)	Lignin (%)
Untreated fiber	47.30	31.85	22.70
Alkalized fiber	56.74	19.30	22.21
Bleached fiber	62.82	18.28	17.90

color during the purification process. After the bleaching process, the color of the fibers turned to a brighter yellowish color compared to the previous treatment of alkalization. Non-cellulosic material such as hemicellulose and lignin content were degraded during the purification process, as shown in **Table 2**.

Morphological Anaysis

The picture resulted from SEM observation showed that the fiber diameter has decreased after the bleaching process compared to the raw material (**Figure 3a**). **Figure 3b** showed the fibers diameter reduction and fibrillation in micrometer size after the bleaching treatment from a diameter of 450±3 µm to a diameter of 350±2 µm.

The mechanical stirring reduces the fiber size to reach a micrometer and nanometer scale. The mechanical stirrer was used as a modification tool with a maximum rotation speed of 20,000 rpm. The rotation speed of 5,000, 10,000, and 15,000 rpm were varied to determine their effect on fibers' crystallinity after processing. The fibrillation of cellulose microfibers after processing by a mechanical stirring is shown in **Figure 4**.

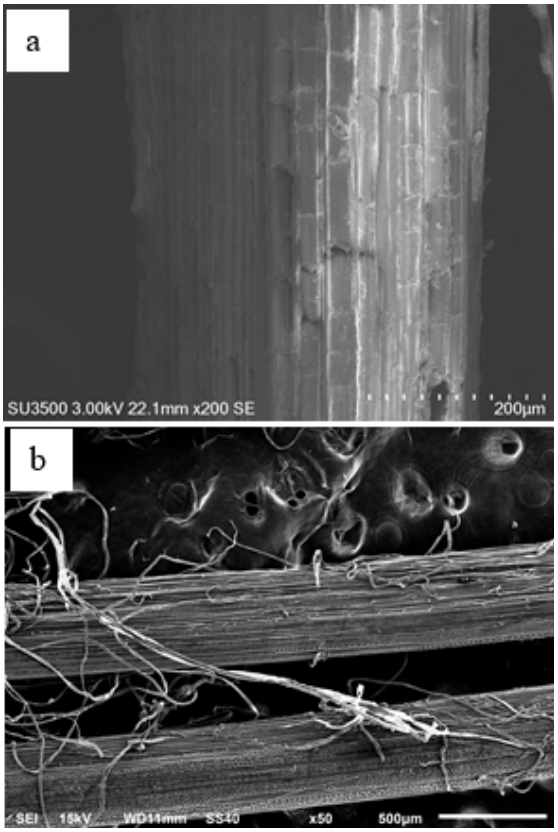

Figure 3. SEM micrograph of a) raw fiber and b) bleached fiber

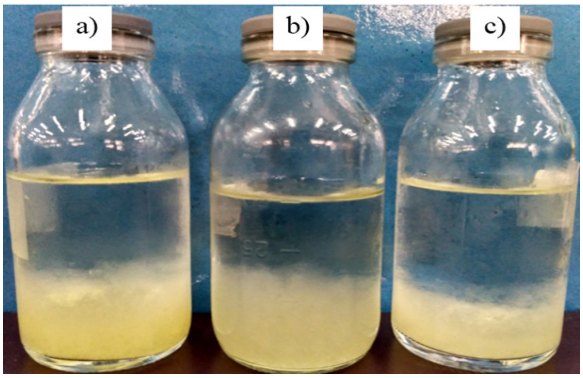

Figure 4. CMF after processing by mechanical stirring for: a) 5,000, b) 10,000 and c) 15,000 rpm

Figure 5 shows the SEM images of CMF from the M5000 sample with the highest crystallinity index (CrI). The cellulose microfiber showed a broad polydispersity of 100-300 µm in length (**Figure 5a**), 5-10 µm in diameter (**Figure 5b**), and 10–25 in aspect ratio. These results are similar to previous research by Maheswari *et al*. (2012) on Coconut palm fiber leaf sheath with Chlorination and alkalization that produced cellulose microfiber with a diameter of 10-15 µm.

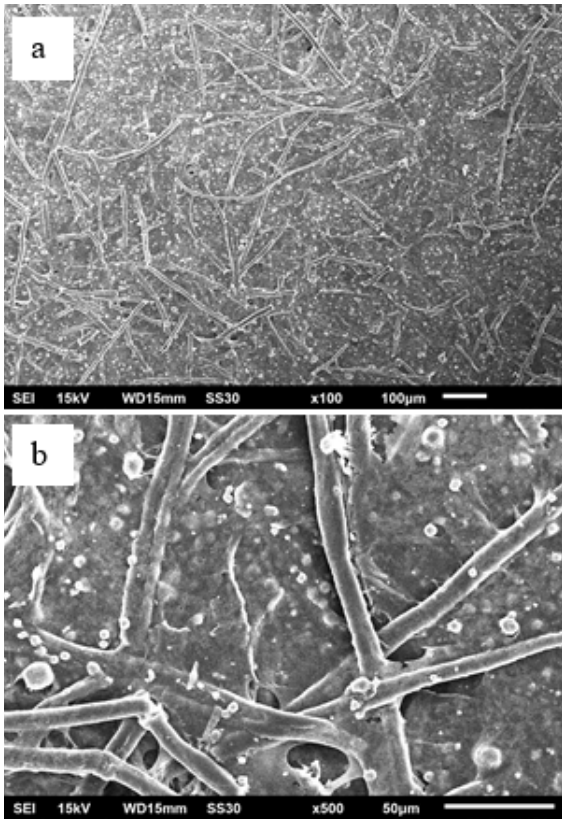

Figure 5. SEM image of CMF from the M5000 sample: a) 100x and b) 500x magnification

XRD Analysis

Figure 6 shown the XRD spectra of raw fiber, alkalized, and bleached fiber. All samples showed characteristic peaks of cellulosic fiber at 16.4°, 22.5°, and 34.8°, corresponding to the crystallographic plane of (110), (002), (040), respectively (Maheswari *et al*., 2012). According to Mohammed *et al*. (2015), these peaks are characteristic of crystal structure for cellulose I on the XRD results of bamboo fibers. The pattern consists of crystalline and amorphous peaks, which are types of semicrystalline material (Tonoli *et al*., 2012). Hemicellulose and lignin-based in these observations, contribute to the amorphous material (Ferrer *et al*., 2016). These patterns Purification of cellulose consisted of alkaline and bleaching, due to non-cellulosic materials were degraded, each treatment showed an increase in the value of the crystallinity index. The crystallinity index of the raw fiber after the alkalization and bleaching process is presented in **Table 3**. The increase in the crystallinity index indicates that the amorphous area decreases after chemical treatment (Sonia and Dasan, 2013).

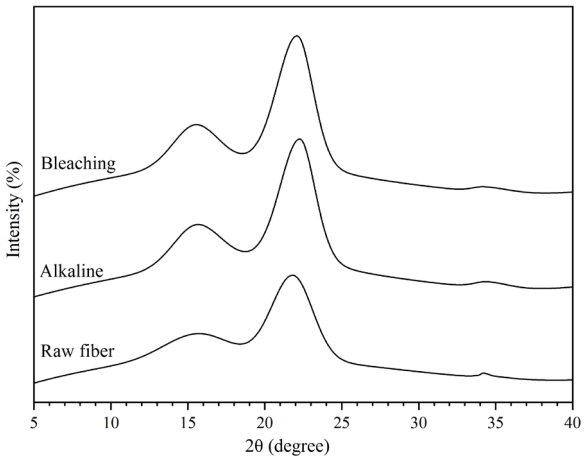

Figure 6. XRD pattern for the cellulose purification process

Table 3. Crystallinity index each step for cellulose purification

Sample	Crystallinity Index (%)
Untreated fiber	64.3
Alkalized fiber	67.7
Bleached fiber	70.4

Microfiber aggregation with hydrogen bonds in the fiber framework is affected by the hemicellulose content. The physic of hemicelluloses will fill the gaps between the cellulose microfibrils. They will act as a physical barrier that inhibits damage caused by the blade shear forces. (Chaker *et al*., 2013). XRD testing was carried out on all samples of the mechanical stirring process to analyze its effect on the fiber crystallinity index. Similar to the research results by Panthapulakkal and Sain (2012), the crystallinity index after mechanical treatment was higher than the previous treatment. The three patterns show crystal planes corresponding to 110 and 002 at the peaks of 16˚ and 22˚, respectively. The obtained crystallinity indexes were determined by Segal's equation. The crystallinity index of cellulose microfibers did not change substantially with stirring rotation speed and time. The crystallinity index values were estimated at 77.4%, 78.2%, and 79.1% for the M15000, M10000, and M5000, respectively. The high-speed rotation at 15,000 rpm for 30 minutes has affected the crystallinity index. The shear of the blade at high temperature (65^{o}C) on the cellulose affects the decreasing of CI. The

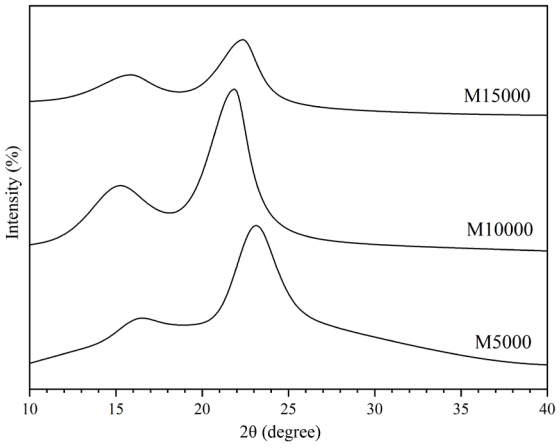

Figure 7. XRD pattern for the mechanical treatment

high temperature of cellulose can damage the crystalline structure due to high friction during the mechanical stirring process.

The low-speed rotation at 5,000 rpm for 90 minutes shows the highest crystallinity index. It does not affect CI because low friction results in a low temperature process (45°C).

Thus, the chemical and mechanical treatments affect the crystallinity of cellulosic material (Ferrer *et al*., 2016). **Figure 7** shows the X-ray diffractograms of all samples after mechanical treatment. Finally, we noted that to produce cellulose microfiber, the mechanical treatment with low-speed rotation (5,000 and 10,000 rpm) did not significantly affect the crystallinity index, which was confirmed from the analysis results of the XRD test. Similar to the research of Sofla *et al*., (2019), isolated cellulose from bagasse using a high-speed blender with medium speed and a long-time process (60 minutes) did not substantially affect the change in the crystallinity index of CNF materials. The ratio of cellulose and water used was 1:1,000, which aims to reduce the excessive friction between the cellulose and the blade and cause the temperature process to stabilize (50-60°C).

FTIR Analysis

The spectroscopic approach is one of the methods used to characterize cellulosic fibers. FTIR spectroscopy was used to identify the effect of mechanical stirring on functional groups within the fibers. The FTIR spectra of raw fiber, bleached fiber, and cellulose microfibers are shown in **Figure 8**.

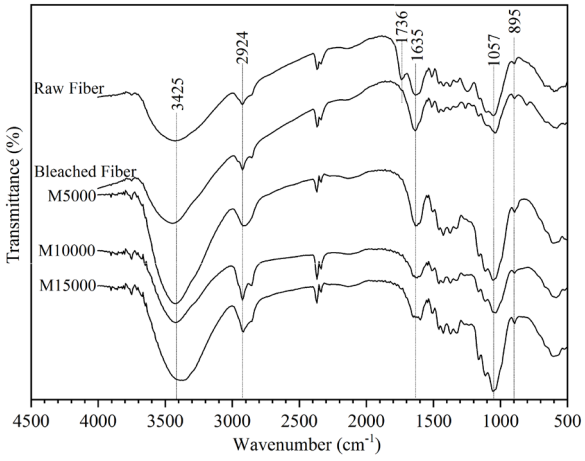

Figure 8. FTIR spectra of raw fiber, bleached fiber, and cellulose microfiber

All samples presented cellulose, hemicellulose, and lignin with their typical vibration bands. A strong absorption band at about 3,425 cm^{-1} is intermolecularly bonded hydroxyl (O-H) groups (Reddy *et al*., 2012). The broad bands at 2,924 cm^{-1} correspond to the C-H stretching of methyl and methylene groups in cellulose, hemicellulose, and lignin (Maheswari *et al*., 2012). The peak at 1,736 cm^{-1} seen in the raw fiber disappeared upon alkaline treatment. This is because the carboxylic group was removed by an alkali treatment called the deesterification process (Mwaikambo *et al*., 2002). The peak at 1,635 cm^{-1} is the absorbed water in cellulose (Jankwoska *et al*., 2020). The peak at 1,504 cm^{-1} assigned to the C=C stretch of the aromatic ring was taken as a lignin reference (Chaker *et al*., 2013). The stretching (C-O) and rocking (C-H) vibration of cellulose occurred at the absorbances of 1,057 and 895 cm^{-1} (Ferrer *et al*., 2016).

Conclusion

The chemical treatment succeeded in reducing the non-cellulose content of the salacca midrib fiber. The presence of non-cellulose content that is still attached to the cellulose can prevent damage to the crystal structure due to friction during the high-speed stirring process. The rotation speed during the mechanical stirring process affects the crystallinity index more than the operation time. The highest crystallinity index value was 79.09% for sample M5000. The average fiber diameter resulted from the isolation of cellulose

microfibers by mechanical stirring ranged from 10-15 μm with a length of 100-300 μm.

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