

Article

Silica Scaling Inhibition in Water Treatment Process Using Fibrous Al₂O₃-Nylon 6 Adsorbents

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Abstract: This study describes a novel approach using fibrous Al₂O₃-Nylon 6 composites to induce inhibition behavior in silica scaling systems. The composite fibers were fabricated with a wet-spinning process using the coagulation of a methanolic Nylon-CaCl₂ solution with Al₂O₃ powder after immersing the thread-like solution in water. The mesoporous nylon fibers composed of Al₂O₃ powders ranging from 10 to 30 wt% loading demonstrated superior adsorption capabilities to silica in water, behaving with the Freundlich model and exhibiting effective multilayer adsorption onto the Al₂O₃ sites embedded in the fiber. Furthermore, the composite fibers inhibited silica scaling, even at high concentrations, due to a substantially efficient reduction in soluble silica when the composite fiber was present in the system. The utilization of 15 g of composite fibers resulted in a rapid drop to approximately 30 mg/L within the initial 10 h, which is a considerable improvement compared to the 300 mg/L observed in the fiber-free control sample. Notably, the presence of an elevated fiber content exceeding 7.5 g demonstrated the complete inhibition of silica precipitation. An analysis of the pore volume using nitrogen adsorption experiments before and after silica adsorption showed that silica adsorption resulted in a significant decrease in mesoporous properties at the alumina sites. This indicated an efficient adsorption of silica onto the alumina site, effectively removing silica from the system.

Keywords: silica adsorption; silica-scaling inhibition; wet-spinning process; activated alumina; Nylon 6



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1. Introduction

Silica scaling represents a practical problem in water systems and becomes a substantial challenge within cooling systems [1,2], geothermal power plants [3,4], and reverse osmosis (RO) systems [5–7]. The typical concentration of silica in natural waters ranges from 5 to 60 mg/L, occasionally peaking at 300 mg/L in some volcanic areas [8,9]. When the concentration of silica exceeds its solubility limit, typically falling within the range of 100–150 mg/L at a near-neutral pH [10,11], an excess of silica becomes insoluble and then precipitates, fostering the development of scale. In these cases, many factors affect the process of silica scaling, including pH, temperature, ionic strength, and the interaction with other ions present in tap water [12]. The formation of a stable colloidal suspension silica unfolds in three key phases: supersaturation, nucleation, and growth [13]. Silica is typically deionized at natural pH levels and initially exists in a soluble monomeric form referred to as mono-silicic acid (Si(OH)₄). However, as concentrations increase, mono-silicic acid undergoes rapid polymerization, giving rise to polysilicic acid, which is dehydrated to form Si-O-Si anhydride bonds [6]. This condensed process leads to the progress of further scaling and to the formation of larger silica species, ultimately manifesting as colloidal forms. Consequently, this phenomenon significantly compromises operational efficiency,

leading to clogging in the water flow, reducing the lifespan of water transportation systems, and necessitating an increase in chemical and mechanical operational costs to recover the system [12–14].

To date, numerous strategies have been proposed to address silica scaling by using chemical inhibitors (e.g., lime, caustic soda, and soda ash) [15–17], pH adjustments [18], and ion exchange [19], precipitation [20], softening [21], and coagulation [22] methods. However, these approaches still lack practicality due to insufficient silica removal and the generation of significant sludge, resulting in shortcomings in terms of cost-effectiveness, environmental impact, or sustained efficacy over time [6,23]. As the demand for efficient and environmentally friendly solutions grows, there is a compelling need to explore alternative approaches to silica mitigation. Among these, silica adsorption onto aluminum compounds [6,24], particularly activated alumina [23,25,26], was reported as a crucial adsorbent due to its high specific surface area and mesoporous properties.

Despite the above-mentioned advantages for Al_2O_3 adsorbents, the use of activated alumina in its powder-stated initial absorbents poses some drawbacks in terms of low permeability in the post-adsorption process, with pressure drops in the continuous treatment of the water flow [27]. To overcome this obstacle, as an alternative to a powdered adsorbent, polymer–inorganic composite fibers were used to create less of a pressure drop for water permeability [28,29]. Fibrous polymer–inorganic powder composites have the advantage of being porous and having a large adsorption area. Therefore, fibers with activated alumina composites in a fibrous, porous polymer matrix can be expected to efficiently adsorb silica. In previous fibrous materials, nylon-based inorganic composites were widely employed with various inorganic fillers, using the electrospinning method to produce boehmite nanofibers and crystalline fibers [30] with excellent mechanical, thermal, and chemical properties [31–34]. Besides its effectiveness in producing high-quality nanofibers, electrospinning faces challenges in large-scale production due to a limited production rate and the complexity of its setup and operation [35]. On the other hand, the fabrication of fibrous inorganic polymer composites using wet-spinning coagulation processes gives them potential for water treatment adsorbents. In particular, the advancement of zeolite–polyethersulfone composite fibers through wet-spinning methods demonstrated their effectiveness in practical applications for the decontamination of radioactive cesium [28], contributing to the recovery of radioactive cesium in Fukushima, Japan. Moreover, these composite fibers were also utilized for heavy metal ion removal [29], organic dye bleaching [36], and degasification to toxic H_2S in soil [37]. These successful processes have the following advantages: inorganic powders are embedded in porous polymer fibers, showing that inorganic powder function remains in the fiber. Nevertheless, there are no reports on the utilization of Nylon 6 in the production of composite fibers prepared with a wet-spinning method, primarily owing to the lower solubility associated with Nylon 6 in solvents. Interestingly, there are limited reports available regarding the fabrication of Nylon 6-based zeolite composite membranes designed especially for the selective adsorption of heavy metal ions [38] and ethylene gas [39] by dissolving Nylon 6 in $\text{MeOH}/\text{CaCl}_2$. However, even though such a solvent in the wet-spinning process is applied to Nylon 6-fibers, there is high potential to find optimum conditions. This is a challenge in developing novel Nylon 6 composite fibers.

With this basic background, in the present study, novel Nylon 6 composite fibers with alumina powders were fabricated using a $\text{MeOH}/\text{CaCl}_2$ solvent for the wet-spinning coagulation process, and they were then applied for silica elimination in water to address the issues of a lower scaling process. Silica adsorption experiments were conducted to investigate the adsorption characteristics of silica, and they focused on addressing the inhibition of silica scaling at high silica concentrations, aiming to solve scaling in desalination and water treatment processes.

2. Results and Discussion

2.1. Fabrication of Al_2O_3 -Nylon 6 Composite Fibers

Figure 1 described the fabrication of Al_2O_3 -Nylon 6 composite (ANC) fibers using a wet-spinning method in a $\text{MeOH}/\text{CaCl}_2$ solution [38]. Firstly, a $\text{MeOH}/\text{CaCl}_2$ solution was prepared with 15 wt% of CaCl_2 dissolved in methanol. Then, 20 wt% Nylon 6 granules was introduced to the solution and vigorously stirred at 60 °C until the granules dissolved completely. The resulting viscous Nylon 6 solution was combined with activated alumina powders of 10, 20, and 30 wt% in loading to Nylon 6, referred to as ANC 10, ANC 20, and ANC 30, respectively (Figure 1). This mixture was then extruded into a cylindrical container fitted with a hollow needle at the bottom, and a pressure of 0.6 MPa was applied. When the solution was extruded through the needle into a substantial volume (5 L) of deionized water, methanol was dissolved into the water, leading to the solidification of Nylon 6 into a fibrous shape through the incorporation of alumina powder. After that, the fibers were washed with deionized water to remove residual MeOH , and finally, the Al_2O_3 -Nylon 6 composite (ANC) fibers were dried under a vacuum. As shown in Figure 1, all obtained fibers displayed a white coloration. The white composite fiber spinning process exhibited the ability to produce circular cross sections and fibrous precipitate in water. Scanning electron microscope (SEM) images of the resultant fibers for the surface and cross section are also displayed in Figure 2. In the cross-sectional analysis, the diameter of the fibrous Nylon 6 matrix, both with and without alumina, measured approximately 500 μm in each fiber. The Nylon 6 formed a fine spongy network of about 1 μm , observed within the interior of the fiber. The morphology of the cross section became denser with the increasing alumina powder loading. Examining the morphology at $\times 3000$ magnification of Nylon fiber and ANC fibers revealed a consistent distribution of Al_2O_3 powder within the Nylon fibrous matrix.

Table 1 provides the mechanical properties of the composite fibers. The tensile strength values of the fibers tended to decrease when the Al_2O_3 loading increased from 0% to 30 wt%. The density of Nylon fiber was 1.02 g/cm^3 , along with tensile strength and elongation values of 8.6 MPa and 15.5%, respectively. In comparison to ANC 10, ANC 20, and ANC 30, the values of density increased from 1.54 to 1.67 g/cm^3 with an increase in the Al_2O_3 content. The elemental composition of the composite fibers, as revealed in Table 1 through an XRF analysis, further supports this observation. The Al_2O_3 content (wt%) was measured at 11.2%, 23.2%, and 32.3% for ANC 10, ANC 20, and ANC 30, respectively. This indicated that the amount of Al_2O_3 deposited on the ANC composite fiber was proportional to the dose-distribution of the Al_2O_3 loading amount. Additionally, the residual CaCl_2 additive in the composite fibers was taken into consideration. The percentages of calcium within Nylon 6, ANC 10, ANC 20, and ANC 30 were 0.009%, 0.044%, 0.062%, and 0.103%, respectively. Likewise, the percentages of chloride were 0.083%, 0.076%, 0.061%, and 0.038% for Nylon 6, ANC 10, ANC 20, and ANC 30, respectively. Notably, the percentage of calcium within the ANC fibers exhibited an increase with the loading of Al_2O_3 due to the activated alumina powder containing 0.013% calcium. However, the component derived from CaCl_2 was negligibly small compared to the Al content, suggesting that washing with water was efficient. The results of the evaluation of fiber porosity via nitrogen adsorption are also included in Table 1. The nylon fiber exhibited a surface area of 7.0 m^2/g , whereas the ANC fiber demonstrated a significantly increased surface area of 151.2 m^2/g , indicating a dramatic increase. The introduction of Al_2O_3 to the fiber matrix, as seen in the BET surface area analysis, created a substantial porous structure. With the increasing Al_2O_3 loading, the fiber featured mesopores with a pore size of approximately 3 nm and a volume of about 0.13 cm^3/g for ANC 30.

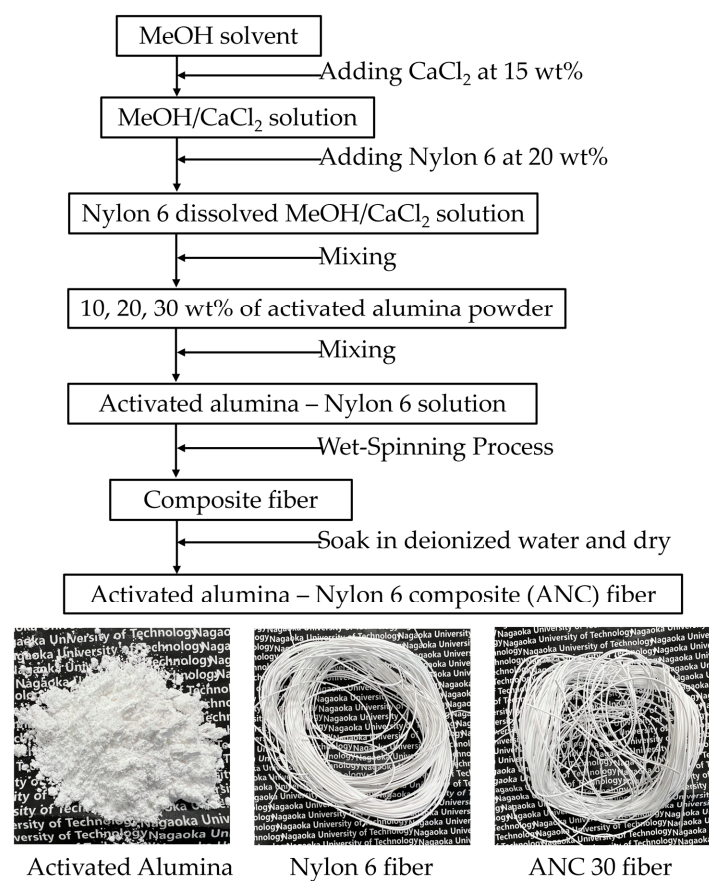


Figure 1. Fabrication procedure of Al_2O_3 -Nylon 6 composite fibers.

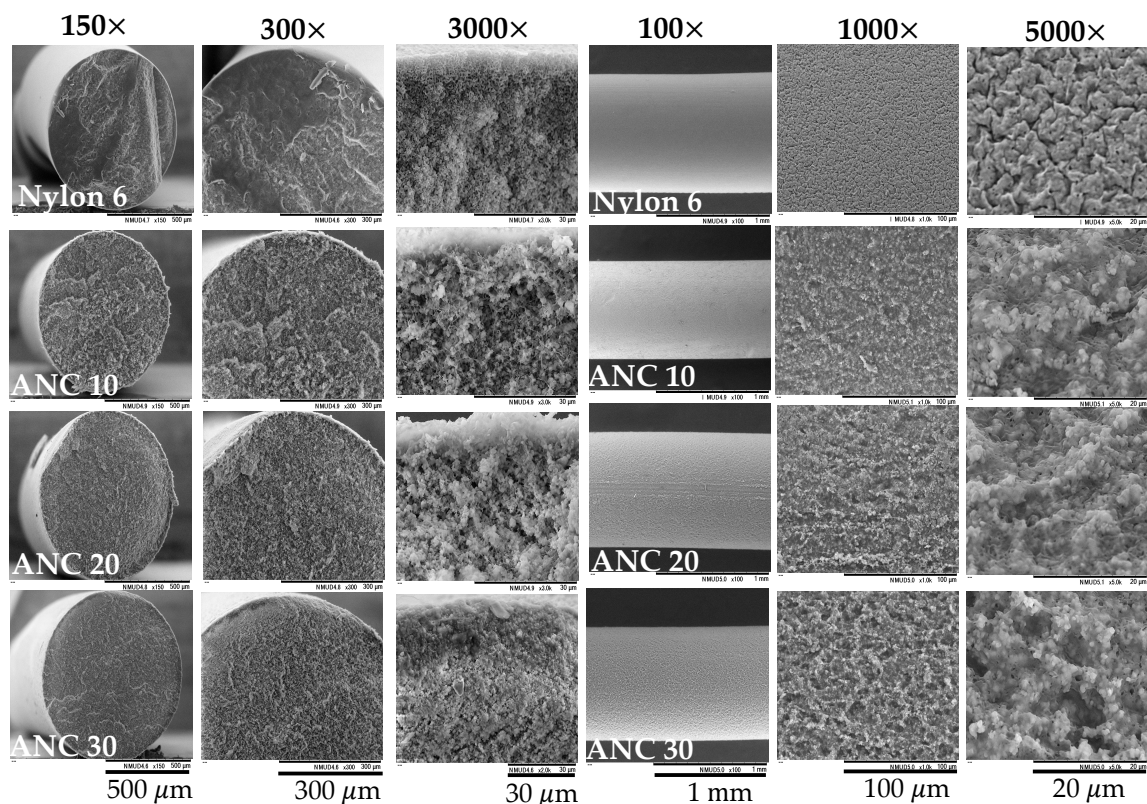


Figure 2. SEM images for cross-sectional area (left) and surface area (right) of Al_2O_3 -Nylon 6 composite fibers.

Table 1. Properties of Al₂O₃-Nylon 6 composite fibers.

	Al ₂ O ₃ Content (wt%)	Tensile Strength (MPa)	Elongation (%)	Density (g/cm ³)	BET Surface Area (m ² /g)	BJH Pore Volume (cm ³ /g)	BJH Pore Size (nm)	Elemental Composition		
								Al	Ca	Cl
Al ₂ O ₃	100	-	-	-	217.1	0.21	3.27	44.4	0.013	-
Nylon 6	0	8.6 ± 0.4	15.5 ± 1.5	1.02 ± 0.05	7.0	0.02	5.42	-	0.009	0.083
ANC 10	10	5.4 ± 0.7	9.9 ± 0.9	1.54 ± 0.04	84.7	0.07	3.45	11.2	0.044	0.076
ANC 20	20	3.8 ± 0.9	6.8 ± 0.2	1.63 ± 0.03	125.8	0.11	3.30	23.2	0.062	0.061
ANC 30	30	1.9 ± 0.3	3.8 ± 0.1	1.67 ± 0.05	151.2	0.13	3.25	32.3	0.103	0.038

2.2. Silica Adsorption Behavior of Al₂O₃-Nylon 6 Composite Fibers

To investigate the adsorption of silica onto these fibers, they were immersed in a solution with a silica concentration of 20 mg/L, and the amount of silica bound was measured, resulting in saturated adsorption (Figure 3a). Consequently, the amount of adsorbed silica tended to increase significantly within the initial 24 h, and gradually reached saturation after 3 days. Clearly, the alumina-free nylon fiber absorbed very little silica, about 1 mg/g. Figure 3b demonstrates the relationship between the silica equilibrium concentration and the amount of silica adsorbed. In this adsorption isotherm, the amount of silica adsorbed increased with the increasing concentration. Notably, ANC 30 exhibited a higher adsorption capacity compared to ANC 10 and ANC 20. This might be due to the increased sorption sites of Al₂O₃ in the composite fibers.

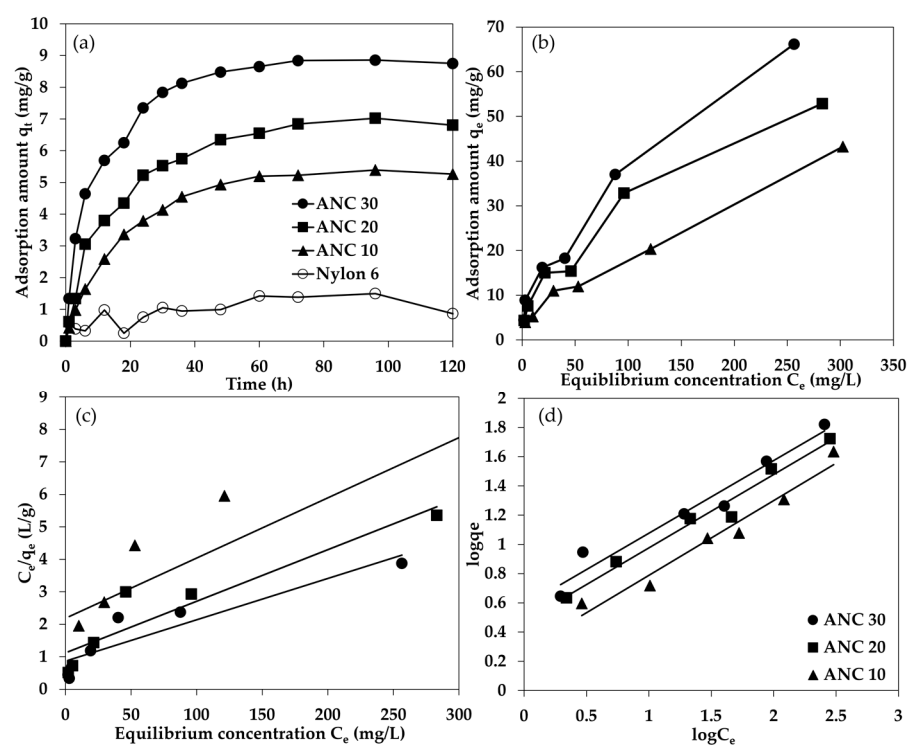


Figure 3. Silica adsorption amounts of composite fibers at (a) different time intervals with 20 mg/L and (b) various concentrations for aqueous silicate at pH 6.5 ± 0.2 ; (c) Langmuir and (d) Freundlich adsorption isotherms.

Figure 3c,d show the plots of the Langmuir and Freundlich models, which were created using the relationship between the C_e of silica in the solution and the adsorption capacity of q_e . The linear coefficients of these equations were used as a basis to elucidate the

mechanism information of the adsorption process in the fiber composites. In the Langmuir models, Equation (1) was utilized for the correlation of adsorption equilibrium.

$$\frac{C_e}{q_e} = \frac{1}{q_m \times K} + \frac{C_e}{q_m} \quad (1)$$

where q_e is the amount of Si adsorbed at equilibrium (mg/g), K is the Langmuir constant, q_{max} is the maximum adsorption capacity (mg/g), C_i and C are the initial and final concentrations of the Si in the solution (mg/L), and V represents the volume of the solutions (L). Freundlich Equation (2) is as follows:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (2)$$

where K_F and n represent the Freundlich constants. As seen from the linearity in the plots of Figure 3c,d, Table 2 provides the coefficients for the Langmuir and Freundlich isotherm models which were applied for the silica adsorption of composite fibers. The results indicated that the Freundlich adsorption isotherm showed a good linear relationship in $R^2 = 0.98$, meaning that the adsorption of Si onto ANC fibers was multilayer adsorption, and the adsorption occurred at specific heterogeneous sites within the adsorbents. Furthermore, the N_2 adsorption isotherms of the Al_2O_3 -Nylon 6 composite fibers displayed a type-IV isotherm in accordance with the IUPAC classification, indicating mesoporous adsorption [40] (Figure 4a). Conversely, the alumina-free nylon fiber displayed type-II isotherms, emphasizing the absence of microporous and mesoporous structures within the fiber [41]. As depicted in Figure 4b, the ANC fibers presented a notable increase in pore volume with a higher Al_2O_3 loading, and the peaks in the pore diameter distribution were observed at about 3 nm, corresponding to the characteristic size of alumina pores. However, when ANC 30 was soaked in 300 mg/L of aqueous silicate for 5 days, there was a noticeable decrease in N_2 adsorption over time (Figure 4c). This remarkable reduction in pore volume indicated that silica adsorption led to the clogging of nm sized pores (Figure 4d). Additionally, the lower value of R^2 obtained in the Langmuir model was 0.83, and the theoretical maximum adsorption capacities were 54.0 mg/g, 63.0 mg/g, and 78.7 mg/g for ANC 10, ANC 20, and ANC 30, respectively. Figure 5 shows the SEM-EDS images of the ANC 30 surface and the cross section after it was immersed in 300 mg/L of aqueous silicate solution for 5 days, highlighting the Al, O, and Si components. Clearly, the cross section of the fiber soaked in silica water shows that the densities of oxygen and silica were increased. This indicates that silica penetrated into the fiber interior and was adsorbed. Comparing the fiber surface images (c and d) showed higher oxygen and silica densities on the surface after immersion, suggesting that silica is also adsorbed on the surface in the form of SiO_2 .

Table 2. Parameters on Langmuir, Freundlich, pseudo-first-order, and pseudo-second-order models.

Isotherm Models	Parameter	ANC 10	ANC 20	ANC 30
Langmuir	K_L	0.008	0.014	0.015
	q_m (mg/g)	54.0	63.0	78.7
	R^2	0.835	0.878	0.873
Freundlich	K_F	1.887	2.975	3.792
	n	1.954	1.990	2.013
	R^2	0.972	0.975	0.955
Pseudo-first-order	K_1	0.046	0.050	0.071
	q_1 (mg/g)	5.2	5.5	6.1
	R^2	0.982	0.983	0.987
Pseudo-second-order	K_2	0.02	0.01	0.01
	q_2 (mg/g)	6.55	8.00	9.64
	R^2	0.997	0.996	0.997

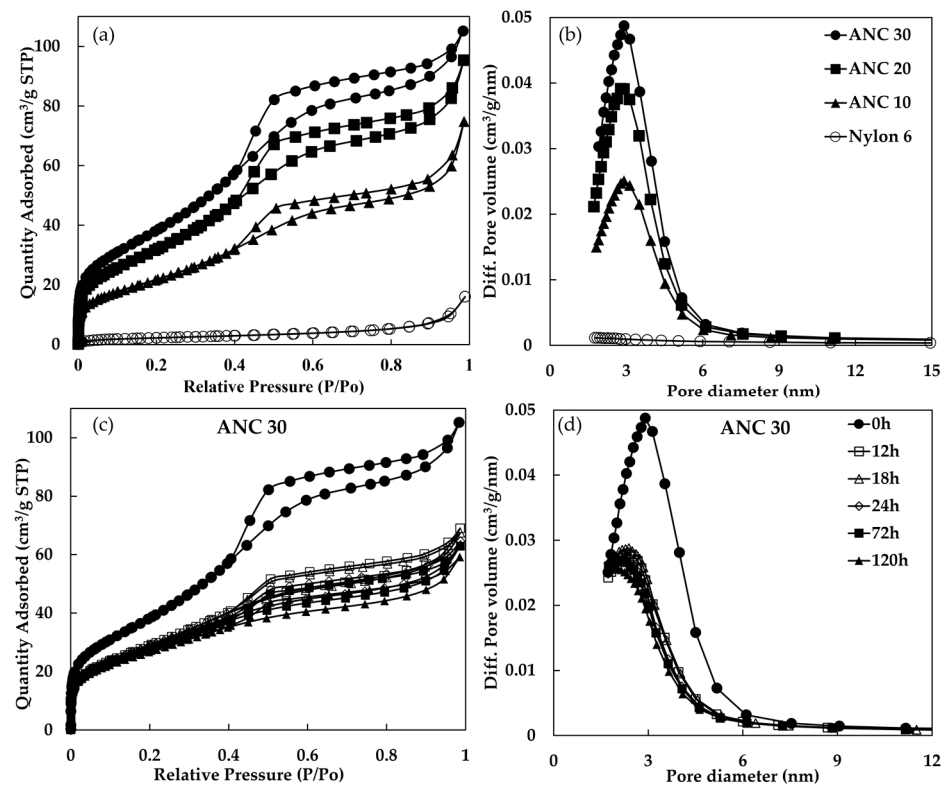


Figure 4. N₂ adsorption isotherms (a) and BJH pore size distribution (b) for Al₂O₃-Nylon 6 composite fibers before adsorption and (c,d) after adsorption for ANC 30 at different immersion times in aqueous silica solution.

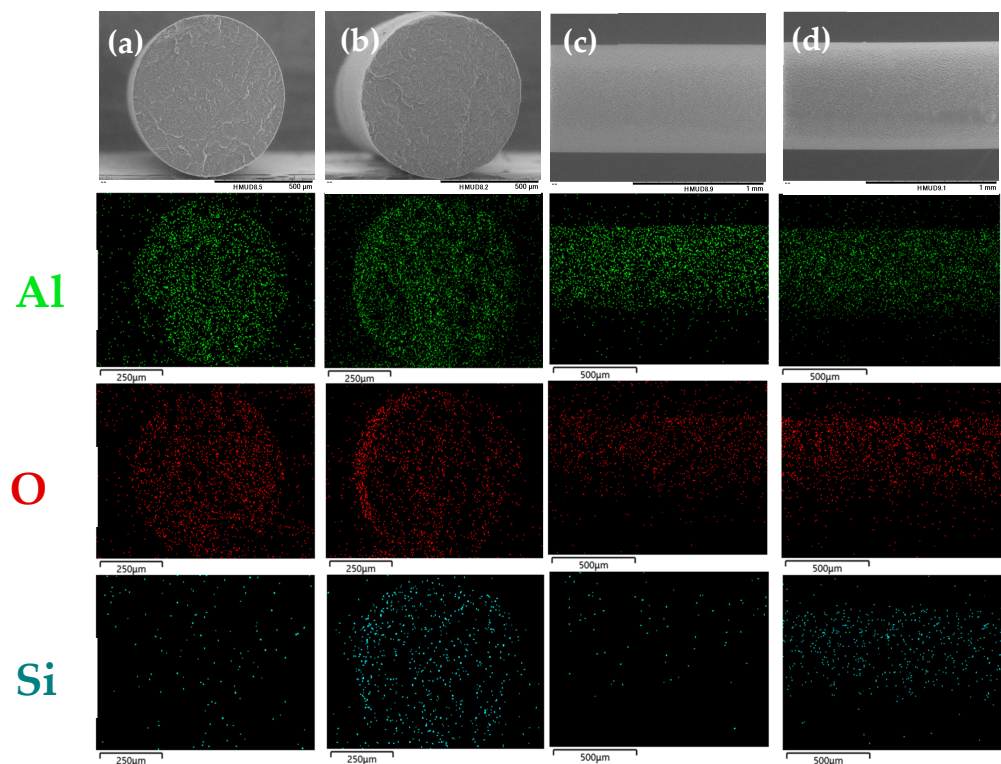


Figure 5. SEM-EDS images of the cross section and surface of ANC 30 before (a,c) and after (b,d) it was immersed in an aqueous silicate solution, highlighting Al, O, and Si components.

To evaluate the effect of contact time on the adsorption rate of silica by the prepared composite fibers, pseudo-first-order (PFO) and pseudo-second-order (PSO) were applied to clarify the adsorption kinetics (Figure 6). The linear forms of PFO (3) and PSO (4) are shown below:

$$\ln(q_e - q_t) = -k_1 t + \ln q_e \quad (3)$$

$$\frac{1}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

where q_e and q_t are the adsorption capacity at equilibrium and at time t (h), respectively. k_1 (1/h) and k_2 (g/mg.h) are the rate constant for PFO and PSO. The most suitable model was selected based on the values of the linear regression correlation coefficient (R^2). As shown in Table 2, the pseudo-first-order and pseudo-second-order kinetic models were both suitable for illustrating the adsorption process of Si on ANC fibers. Notably, the pseudo-second-order model exhibited a higher correlation coefficient value (0.99), implying a significant involvement of chemisorption between Si ions and ANC fibers as the primary adsorption mechanism. Hence, the FT-IR spectra of Al_2O_3 , Nylon 6, and ANC 30 were measured to evaluate the interaction of silica and Al_2O_3 in the ANC fibers. As depicted in Figure 7, the FT-IR bands of the nylon fiber were assigned at 1544 cm^{-1} (corresponding to amide II, C–N stretch, and CO–N–H bend) and 1626 cm^{-1} (amide I, C=O stretch), along with –NH bending at 3091 cm^{-1} and –NH stretching at 3315 cm^{-1} [42,43]. The FT-IR bands of alumina were observed at 630 cm^{-1} (OH–Al=O bend), and the peak at 745 cm^{-1} was attributed to the stretching vibration of Al–O–Al [44]. The bands at 1634 cm^{-1} and 2076 cm^{-1} were attributed to the H–O–H bending vibration of the H_2O molecule, and the two bands with wavenumbers 3312 and 3091 cm^{-1} were attributed to the –OH stretching vibration. Notably, the characteristic bands representing the amide group at 1544 and 1626 cm^{-1} , as well as the –NH vibration band at 3091 and $3315\text{--}3480\text{ cm}^{-1}$ in the FT-IR band of Nylon 6 were clearly seen in ANC 30. Furthermore, the incorporation of Al_2O_3 with the nylon fiber matrix appeared in the broadness band, and the intensity was reduced in the –OH stretching band at 3480 cm^{-1} . After the uptake of silica, the adsorption peak around 1074 cm^{-1} attributed to the symmetric bending mode of Al–OH was caused by broadening, and a shift to 1066 cm^{-1} was noted for the SiOH band [45,46]. Also, the peaks observed at 628 cm^{-1} and 738 cm^{-1} relating to Al–O–bonding was shifted to lower wavelengths of 546 cm^{-1} and 726 cm^{-1} . Thus, the chemical interaction between the ANC fibers and silica was confirmed.

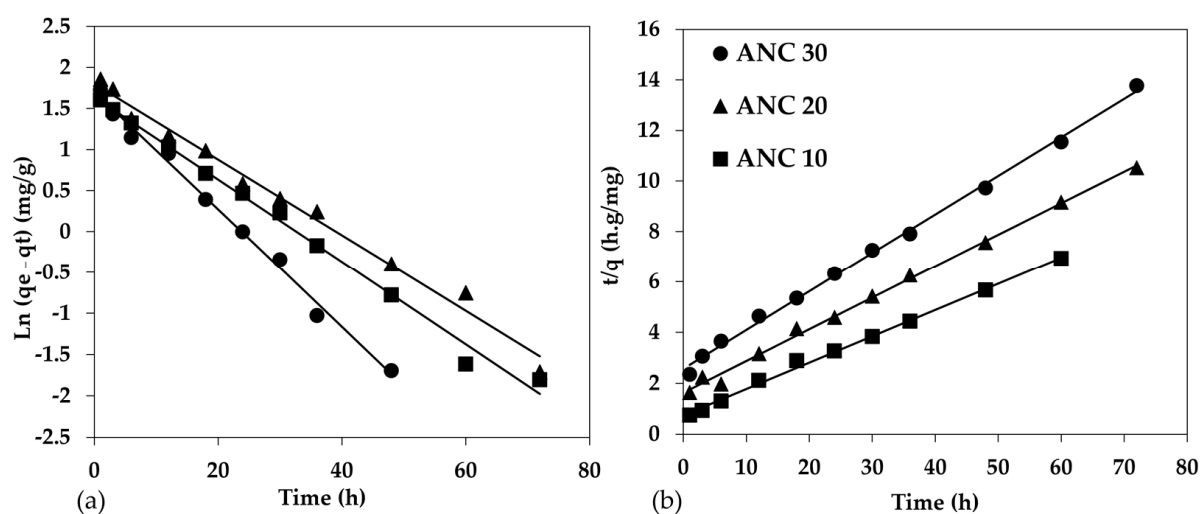


Figure 6. (a) Pseudo-first-order and (b) pseudo-second-order kinetic plots.

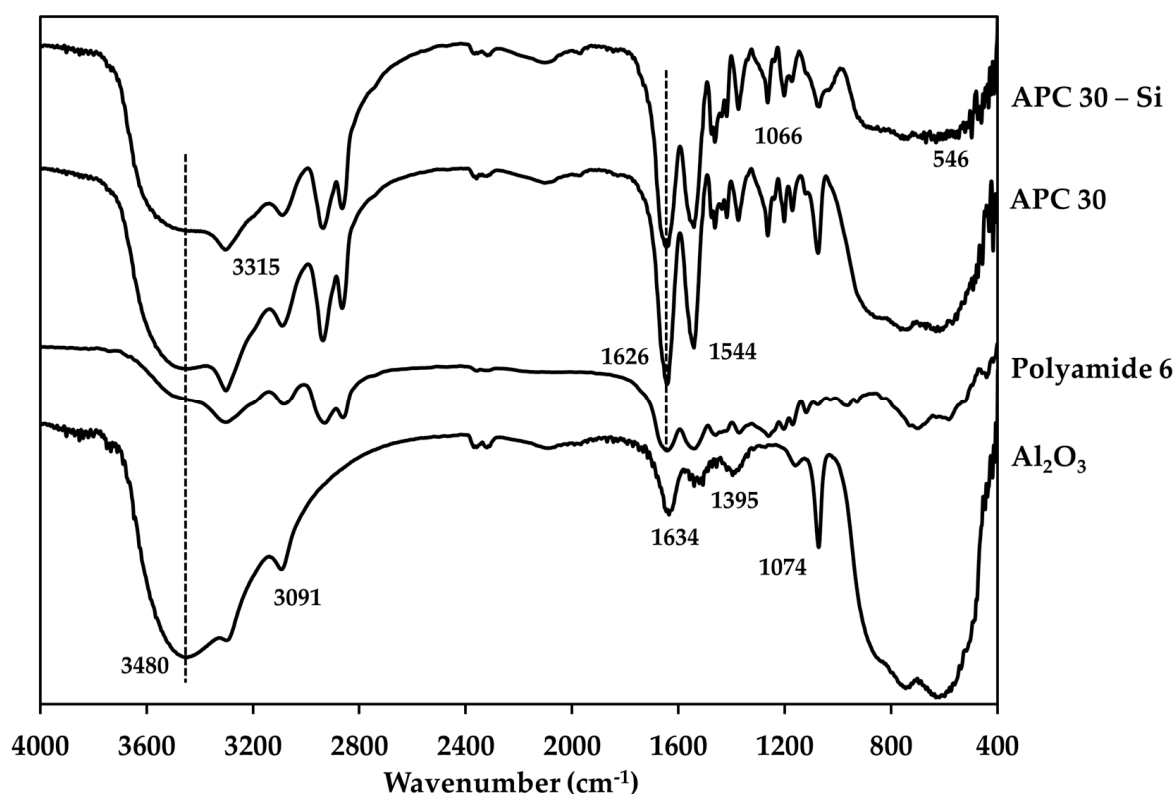


Figure 7. FTIR spectra of Al_2O_3 , Nylon 6, and ANC 30 before and after Si adsorption.

2.3. Silica Scaling Inhibition Effect of Al_2O_3 -Nylon 6 Composite Fiber

Since the results of previous studies showed that ANC 30 with a high alumina content showed good silica adsorption properties, the relationship between the concentration of soluble silica and the amount of ANC 30 fiber was investigated, with Figure 8a showing evidence that a significant silica deposition decreases in the presence of ANC 30 fiber. Notably, there was a sharp decrease in the silica concentration in the solution with increasing amounts of Al_2O_3 -Nylon 6 composite fibers, ranging from 1 g to 15 g, indicating the effectiveness of the fibers in removing silica even at elevated concentrations. Within the first 10 h, the soluble Si content dropped to approximately 30 mg/L when 15 g of fiber was used, whereas the Si concentration remained at 300 mg/L for the control sample without fibers. This trend was further supported by observing the Si precipitation mass, as depicted in Figure 8b. It was noted that after 120 h, the Si precipitate notably increased from 250 mg to 650 mg in the absence of fibers, while only 530 mg, 390 mg, and 112 mg of Si mass were generated in the presence of 1, 2, and 5 g of fiber, respectively. Interestingly, no visible Si precipitate was observed within the initial 9 h when using 5 g of fiber. Furthermore, a higher fiber content, from 7.5 g to 15 g, completely inhibited the precipitation of Si. To examine the size distribution of Si in the presence and absence of fiber, the precipitate was sampled and subjected to particle size analysis spanning 6 to 120 h, as depicted in Figure 9. It was observed that the size distribution of samples containing fibers tended to decrease compared to the sample without fibers. Notably, larger silica particles were evident in the blank condition without fibers after the initial 6 h, with a slight increase observed after 120 h. However, the formation of silica scaling was significantly reduced when fiber loading was at 1 g and 2 g, resulting in smaller Si precipitates after 9 h, with no Si particles recorded at 6 h. Furthermore, a substantial shift in distribution was noted within the initial 12 h, ranging from approximately 30 μm to 500 μm for the blank sample and from 0.01 μm to 2 μm for the sample with 5 g of fiber. The size distributions became more comparable between the sample without added fiber and the one with 1 g of fiber, falling within the size range of 50 μm to 400 μm after 120 h. Following the first 12 h, the size range of 10 μm

to 100 μm shifted towards the 0.01 μm to 2 μm range with an increase in fiber loading from 1 g to 5 g. This observation aligns with the findings of the silica precipitation mass, indicating that Al_2O_3 -Nylon 6 composite fibers have the potential to inhibit silica scaling.

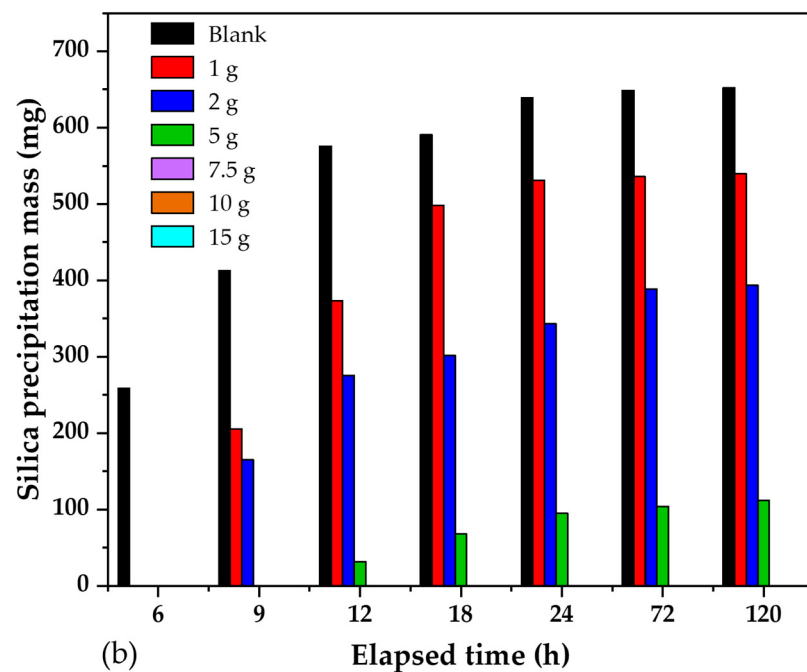
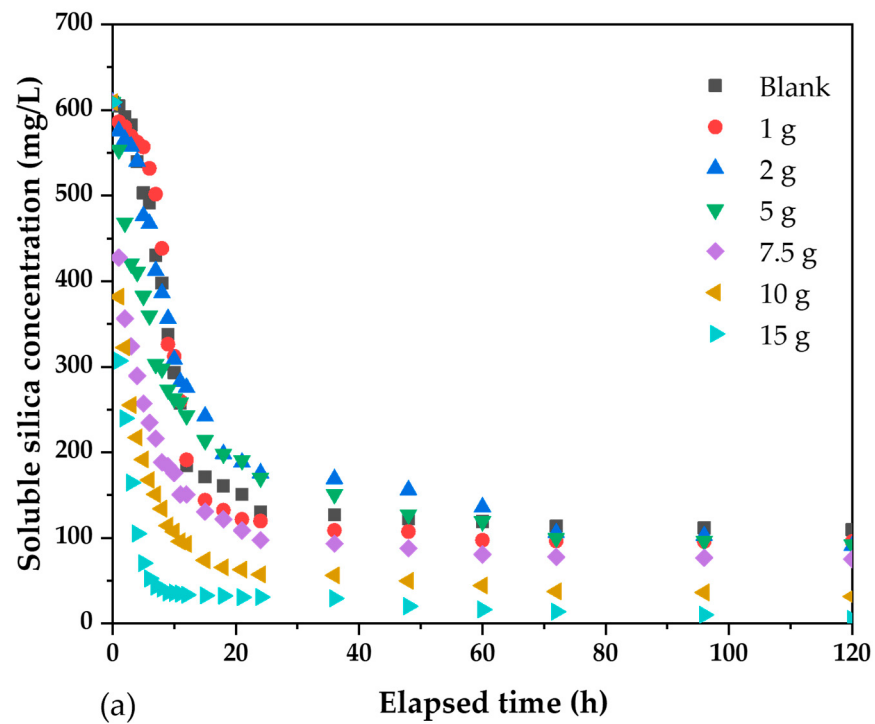


Figure 8. Effect of the content of ANC 30 fiber on the soluble silica concentration (a) and silica precipitation mass (b) for 5 days after an immersion in 600 mg/L aqueous silicate solution.

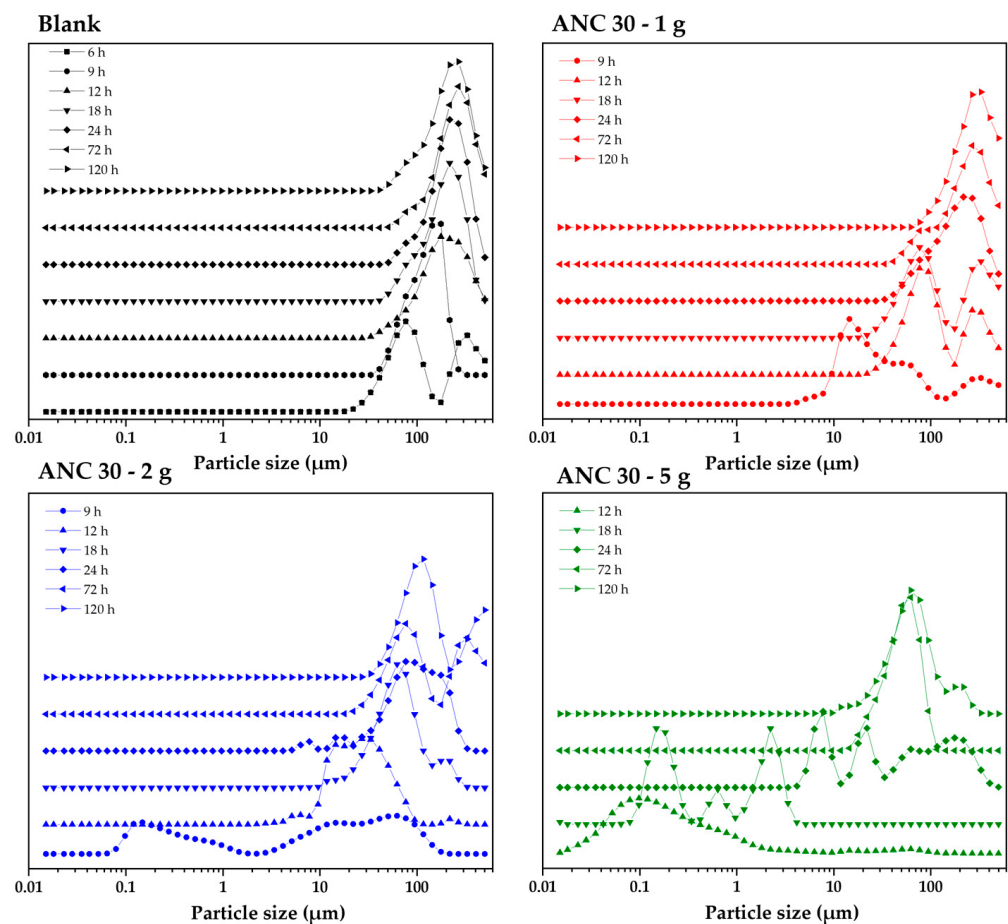


Figure 9. Particle size distribution when incorporating 1 g, 2 g, and 5 g of ANC 30 fiber, along with a blank sample, over a period of 5 days in 600 mg/L aqueous silica solution.

3. Conclusions

The Al_2O_3 -dispersed $\text{MeOH}/\text{CaCl}_2/\text{Nylon 6}$ solution was employed to successfully produce Al_2O_3 -Nylon 6 composite fibers using the wet-spinning coagulation process as the first report for silica adsorption to restrict scaling formation. The Al_2O_3 content within the composite fibers, ranging from 10 to 30 wt%, was uniformly embedded within the Nylon 6 fibers. The silica adsorption experiments revealed that the composite fibers with higher Al_2O_3 contents exhibited higher adsorption abilities due to an increased surface area and mesoporous structure of the fibers. In the equilibrium studies, silica adsorption behavior was followed the Freundlich model, indicating multilayer and heterogeneous adsorption onto the adsorbent in the sites of Al_2O_3 . Furthermore, the composite fibers were evaluated for their effectiveness in inhibiting silica scaling at elevated silica concentrations of 600 mg/L. Within the initial 18 h, the presence of these fibers led to a substantial reduction in silica precipitation. Thus, the presence of Al_2O_3 composite fibers has been shown to have a scale-reducing effect. These findings underscore the potential of composite fibers to effectively inhibit silica scaling, presenting a promising solution for membrane scaling issues.

4. Materials and Methods

4.1. Materials

All chemicals utilized were of analytical grade and required no additional purification. The activated alumina KC-501 powder (Al_2O_3) was obtained from Sumimoto Chemical Co., Ltd., Tokyo, Japan without any further processing. Nylon 6 was sourced from Toray, Industried, Inc., Tokyo, Japan. Methanol (MeOH), calcium chloride (CaCl_2), and various

other chemicals were obtained from Nacalai Tesque, Inc., Kyoto, Japan. The molecular weight of Nylon 6 was determined as 19,000 g/mol as previously described [32]. For the preparation of silica stock solution with a concentration of 1000 mg/L, sodium silicate salt from Fujifilm, Wako, Corp., Osaka, Japan was dissolved in deionized water. The desired silica concentrations were achieved through dilution of this stock solution. When adjusting pH, either NaOH or HCl was utilized.

4.2. Characterizations of Al₂O₃-Nylon 6 Composite Fibers

The morphology of Al₂O₃-Nylon 6 composite (ANC) fibers was observed using a scanning electron microscope (SEM) with an energy-dispersive spectrometer (EDS) (Hitachi, TM 3030 plus, Tokyo, Japan) for inorganic components of Al, O, and Si in both samples before and after the silica adsorption treatment. In the preparation steps, all of the composite fibers were fractured in liquid nitrogen and freeze-dried for 24 h before the gold coating with an acceleration voltage of 15 kV (Sanyu Denshi K.K, Nagoya, Japan), which was used for gold-sputtered samples. X-ray fluorescence (XRF) instrument (ZSX Primus II; Rigaku Corp., Tokyo, Japan) was employed to qualify the percentage of Al₂O₃ and composite fiber components. Surface area analysis and pore size distribution were determined through Bruner–Emmett–Teller (BET) method and the Barrett–Joyner–Halenda (BJH) analysis, respectively, by using a nitrogen gas adsorption instrument (TriStar II 3020; Micromeritics Instrument Corp., Norcross, GA, USA) at 77 K. Tensile strength and elongation for mechanical strength of the composite fibers were assessed with LTS-500 N-S20 equipment (Minebea, Tokyo, Japan). It was conducted with a crosshead speed of 1.5 mm/s and a gauge length of 30 mm by a 500 N head load, at 23 °C, under relative humidity of 50%. Each specimen was 50 mm in length. The diameter of each sample was determined using a micrometer to calculate the cross-sectional area. For each condition, five specimens were tested. Tensile strength and elongation values were calculated using Equations (5) and (6):

$$\text{Tensile strength (N/mm}^2\text{)} = \text{maximum load/cross-sectional area} \quad (5)$$

$$\text{Elongation (\%)} = 100 \times (\text{elongation at rupture/initial gauge length}). \quad (6)$$

Furthermore, the density (ρ) of fibers was calculated using the Formula (7):

$$\rho \left(\frac{\text{g}}{\text{cm}^3} \right) = \frac{W_s}{W_s - W_w} \times (\rho_o - d) + d \quad (7)$$

where W_s is the specimen weight in the air (g), W_w represents the specimen mass in methanol (g), ρ_o is the density of methanol (g/cm³), and d is the density of air (g/cm³) at 20 °C. The Fourier-transform infrared (FT-IR) spectra for Al₂O₃ powder and the composite fibers were recorded using an IRPrestige-21 instrument from Shimadzu Co., Ltd., Kyoto, Japan with 16 scans in the range of 400 to 4000 cm^{−1}, with a spectral resolution of 4.0 cm^{−1}.

4.3. Batch Adsorption Experiment of Silica

Batch adsorption experiments were conducted by immersing the composite fibers in an aqueous silica solution. The Si residual concentration was measured by using Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) (Varian 720-ES; Varian Inc., Palo Alto, CA, USA). The amount of silica adsorbed on the fibers was estimated based on the decrease in silica concentration when the composite fibers were soaked in the aqueous silica solution for 5 days. In particular, standard stock solution (1000 mg/L) was prepared from sodium silicate salt in deionized water at pH 6.5 ± 0.2 and was diluted to the desired concentration from their stock standard solution. Next, the composite fibers (0.1 g) were cut into approximately 2 cm length pieces and were added to 50 mL of the Si solution under a shaken process at room temperature. Then, the fibers were filtered using a membrane filter with a 0.45 µm pore size at different interval times. The uptake amount of

Si to the composite fibers was calculated in remaining supernatant Si concentration using Equation (8):

$$q = \frac{(C_i - C_o)V}{m} \quad (8)$$

where q is the amount of Si adsorbed (mg/g), m is the weight of the adsorbents (g), C_i and C_o are the initial and final concentrations of the Si in the solution (mg/L), and V represents the volume of the solutions (L).

4.4. Scaling Inhibition Effect of Al₂O₃-Nylon 6 Composite Fibers

Under supersaturated conditions, especially at high temperatures, silica precipitation tended to occur, leading to a significant increase in scaling rate [47]; the scaling rate of silica was examined with and without composite fibers. Experiments on silica scaling in the presence of Al₂O₃-Nylon 6 (ANC 30) composite fibers were performed as follows: First, a 500 mL aqueous silica solution with a concentration of 600 mg/L was prepared. The silica solutions were then adjusted to a neutral pH (pH 6.5 ± 0.2) and then subsequently put into a plastic container (1 L volume) with a specific quantity of ANC fibers introduced. A comparative analysis of silica precipitation rates was conducted by preparing a blank sample without composite fibers. Then, the container was incubated at 60 °C, and each day, the precipitated silica was collected from the solution and separated into soluble silica using a centrifuge over a specified time. At the same time with the silica precipitation, the solution was analyzed with ICP measurement to determine the Si concentration. The weight of silica precipitation was measured after drying in an 80 °C oven for 24 h. Additionally, the impact of composite fibers on the particle size distribution of silica precipitation was determined using a laser diffraction particle size analyzer (SALD-7000, Shimadzu Corp., Kyoto, Japan).

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