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Co/Al–Layered Double Hydroxide-Modified Silicon Carbide Membrane Filters as Persulphate Activator for Aniline Degradation

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Abstract: Novel catalytic silicon carbide membrane filters (SCMFs) are synthesized with Co/Al–layered double hydroxide (Co/Al-LDH)-coated silicon carbide powder. After capsuled in a self-designed membrane shell, the SCMFs are utilized in activating persulphate for aniline degradation. Thermal analysis conducted via TG/DTG/DSC examination shows that the heating treatment is beneficial in elevating the activating ability of SCMFs, and the derived Co_3O_4 displays superior catalytical efficiency than Co/Al-LDHs precursor. The XRD patterns and SEM images indicate the sheet-like Co/Al-LDHs are uniformly coprecipitated throughout the surface of SCMFs. Within 20 min, around 95% of aniline is eliminated under 0.7 m of flow velocity and 8:1 of persulphate to aniline ratio. Three-dimensional fluorescence and GC chromatography reveal that distinct by-products exist in the early stage of the aniline degradation process between the sintered and non-sintered Co/Al-LDH-coated SCMFs. The integration strategy of Co/Al-LDH coatings and heating treatment endows traditional SCMFs with robust catalytic properties for engineering-oriented applications in wastewater treatment.

Keywords: silicon carbide; Co/Al-LDHs; persulphate activation; long-term evaluation; aniline degradation

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1. Introduction

In the past decade, silicon carbide (SiC) material has attracted increasing attention in the domain of sensors, electronic chips, microreactors and energy exchangers [1]. Attributed to exceptional physical–chemical properties like high hardness, and chemical stability as well as superior resistance to corrosion and fouling, SiC has emerged as a leading material in the development of advanced inorganic membrane technologies [2,3]. In comparison to the traditional inorganic or organic membranes made from aluminum oxide or high-molecular polymers [4], the SiC-based inorganic membranes have played a transformative role in solving the long-standing challenges in industrial separations under various harsh conditions [5], such as extreme acidity or alkalinity environment, high content of HF acid, high-lipid oily substances, and high concentrations of organic solvents. Regarding the extraordinary performance and properties [2], SiC-based membranes are considered the next generation of filtration materials.

Recently, SiC-based membranes have been intensively utilized in various wastewater treatment processes [3,4]. In spite of the outstanding filtration performance, it should not be omitted that the removal of pollutants by inorganic membranes primarily relies on physical separation [5–7]. This measure does not fundamentally eliminate the contaminants but concentrates them, leaving their potential environmental impact unaddressed. To date, a variety of modification measures are proposed to elevate the filtration efficiency [6–8]; however, a rare modification process is conducted on SiC-based membranes to endow a degradation function for decontamination of pollutions involved in wastewater. Layered double hydroxides (LDHs) [9] are a group of anionic clays that possess a unique layer structure constructed with abundant hydroxyl groups, diverse anionic groups and optional metallic elements, making them highly designable, flexible and stable. Due to their exceptional physicochemical properties and ease of synthesis, LDH-related substances are widely utilized as absorbent and ion-exchange mediums in environmental remedies via absorption and desalination [10]. Specifically, Co-based layered double hydroxides (LDHs) demonstrate significant catalytic potential. For instance, Qi et al. [11]. and Fan et al. [12]. have highlighted the versatile applications of Co-based LDHs in environmental remediation, noting their robust oxidative capabilities which facilitate the breakdown of organic pollutants in aqueous solutions [11,12]. Furthermore, Co-based LDHs are not only solely utilized as catalysts or catalytic precursors in activating normal oxidants like O_3, H_2O_2 and persulphate (PS) for the degradation of pollutions [13], but are also widely employed as coating materials to endow a catalytic function for granular carbon, SiO_2 microspheres and aluminum oxide chips [14]. In spite of solely or coated utilized, the Co-based LDH materials activate oxidants by facilitating electron transfer processes, enhancing the generation of radical species that are effective in degrading persistent pollutants.

On the other hand, current advanced oxidation processes (AOPs), such as Fenton [10], ozonation [11] and photocatalytic oxidation [8,9], are facing significant limitations for aniline degradation. In spite of the effectiveness in degrading aniline, traditional AOP softening involves high costs and energy demands and can generate toxic by-products. Qi et al. [11] indicated that Co-based layered double oxides as catalysts in ozone treatment presented a promising alternative with lower environmental impact. However, a rare study has been conducted to modify SiC materials as catalytic membranes with Co-based LDHs for the degradation of aniline. To extend the catalytic function of SiC-based membranes for wastewater treatment, in this work, Co/Al-LDH-coated silicon carbide membrane filters (SCMFs) were first synthesized and applied as persulphate activator for the degradation of aniline. The characteristics including thermal properties, morphology and mineralogy were scrutinized, meanwhile, the effect of operation conditions and long-term efficiency were evaluated.

2. Materials and Methods

2.1. Materials and Reagents

The fine SiC powders (with diameter of 5~20 μm) were obtained from JMFilltec Co., Ltd., Jiangshan, China. The reagents of ACS reagent grade, including aluminum chloride ($AlCl_3 \cdot 6H_2O$), cobalt chloride ($CoCl_2 \cdot 6H_2O$), sodium hydroxide (NaOH), sodium carbonate (Na_2CO_3), persulphate sodium ($Na_2Si_2O_8$), aniline(C_6H_7N) and diluted hydrochloric acid (HCl, 1 mol/L), were purchased from Machlin reagent Co., Ltd., Shanghai, China. The simulated air, which was composed of nitrogen (>99.99%) and oxygen (>99.99%), was purchased from Jiangshan Specific Gas Co., Ltd., Jiangshan, China, and Milli-Q ultrapure water (>18.0 MU cm) was utilized for all experiments. In addition, the simulated wastewater was synthesized with 100 mg/L of aniline, and persulphate sodium solution (300 g/L) was employed as an oxidant.

2.2. Synthesis of Co/Al-LDH-Coated SCMFs

The process for the synthesis of Co/Al-LDH-coated silicon carbide membrane filters was referred to Qi's report [11,15] and conducted in JMFilltec Co., Ltd., Jiangshan, China. Specifically, under vigorous stirring conditions, 1000 g ± 5 g of fine SiC powders were initially dispersed in 2000 mL of dilute hydrochloric acid solution as SiC slurry, and then 150 mmol of $CoCl_2 \cdot 6H_2O$ and 50mmol of $FeCl_3 \cdot 6H_2O$ with molecule ratio of Co:Fe = 3:1 was co-dissolved to SiC slurry. Furthermore, the SiC slurry was slowly added into 10 L of a basic solution (1 mol/L of NaOH and 0.1 mol/L of Na_2CO_3). In addition, the temperature and pH of the basic solution were maintained at 70 ± 1 °C and 12.3 ± 0.2 until the SiC slurry was totally added. We stopped stirring and precipitating the sediment at 80 ± 1 °C for 24 h. After the aging treatment, the sediment was separated, washed with water three times, and dried at 60 ± 1 °C. Lastly, the sediment was manufactured as raw SCMFs with specific membrane additives (primarily binder bond), and sintered (non-inert gas protected) at 600 ± 10 °C for 10 h. It should be noted that the raw and sintered SCMFs are denoted as R-LDHs@SCMFs and S-LDHs@SCMFs, respectively. SCMFs, which are directly synthesized fine SiC powders without Co/Al-LDH-coated and non-sintered, were utilized as blank control.

2.3. Self-Designed Membrane Shell and Operation Process

The manufacturing process of industrial-grade membrane filters and shells is introduced in Figure 1. Initially, the SiC powders were synthesized as SiC membrane tubes in a special mold, and then multiple SiC membrane tubes were assembled as SiC membrane filters. The SiC membrane shell was utilized in capsuling the SiC membrane filters and employed for industrial separation usage. In comparison with the industrial-grade membrane tubes and fillers, the Co/Al-LDH-coated silicon carbide was utilized in synthesizing the SCMFs. In reference to the industrial-grade membrane shell, the self-designed membrane shell (Figure 1, denoted in the dotted box) was manufactured to evaluate the performance of SCMFs.

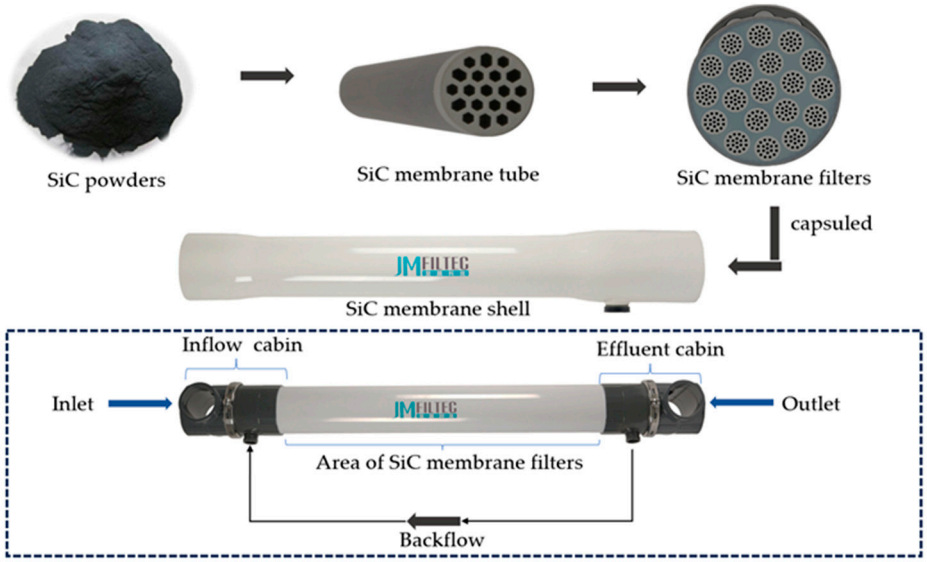

Figure 1. Actual images of SiC powders/membrane tube/membrane filters, and the industrial-grade membrane shell (upper illustrated); and self-designed membrane shell (under illustrated). All images are supplied by JMFilltec Co., Ltd. (Jiangshan, China) (https://a-jmtech.com, rights reserved).

Two operation molds were designed for membrane shells, i.e., batch mold and continuous mold. In this study, the batch mold was activated to evaluate the efficiency of Co/Al-LDH-coated silicon carbide membrane filters, and the operation method was detailed as follows: initially, the membrane shell was filled with the simulated wastewater with a metering pump three times to avoid the absorption effect of SCMFs to aniline. Then, the backflow pump was activated to maintain the flow velocity inside the membrane shell at 0.1–0.9 m/s. Within 0.5 min, a certain volume of persulphate sodium solution was injected one time into the shell along with the backflow. After a certain operation time, the backflow was stopped and the treated wastewater was emptied from the membrane shell. Additionally, the persulphate sodium solution, which was injected into the shell along with the backflow, was maintained at a certain molecule ratio of persulphate sodium versus aniline.

2.4. Characterization

The mineralogy of Co/Al-LDHs that coated SCMFs was certified with XRD, the morphology of silicon carbide powders, R-LDHs@SCMFs and S-LDHs@SCMFs was surveyed via SEM, and the thermal analysis of Co/Al-LDH-coated silicon carbide powders was examined via TG/DTG/DSC. For wastewater treatment, the residue concentration of aniline and by-products is measured by GC chromatography and Three-dimensional Excitation Emission Matrix (3D-EEM) fluorescence. Details of the above-mentioned examining measures and the employed instruments are given in Supplementary Materials S1–S5.

3. Results and Discussion

3.1. Characteristics of SCMFs

The major crystal phases of the R-LDHs@SCMFs and S-LDHs@SCMFs are depicted in Figure 2A. The XRD analysis reveals that R-LDHs@SCMFs primarily exhibit the characteristic peaks at (003), (006), (012), and (110), which are consistent with the diffraction planes of Co/Al-LDH phase (PDF#51-0045). Meanwhile, the XRD pattern of S-LDHs@SCMFs clearly shows peaks of the major phases corresponding to the diffraction planes of cobalt oxide (Co_3O_4) (PDF#73-1701) at (220), (311), (440), and (511). The XRD patterns indicate that the thermal treatment decomposes the coated Co/Al-LDHs as Co_3O_4. This transformation can be attributed to the removal of interlayer water and hydroxyl groups during sintering, as well as the subsequent oxidation of cobalt ions at elevated temperatures [11].

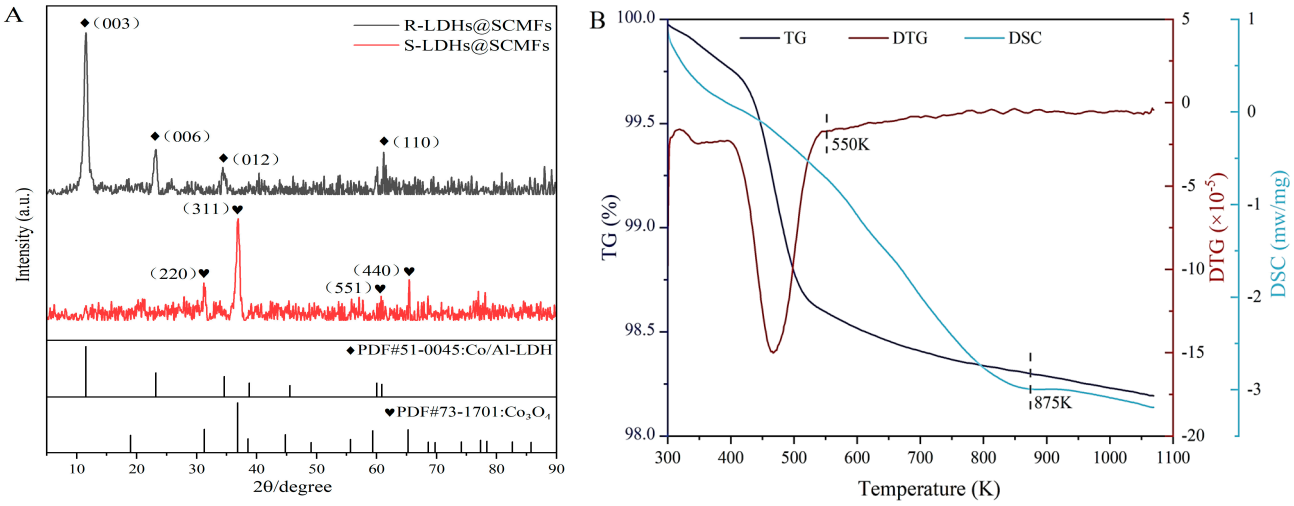

Figure 2. The XRD patterns (**A**) and thermal analysis (**B**) of R-LDHs@SCMFs and S-LDHs@SCMFs.

The thermal analysis of R-LDHs@SCMFs is illustrated in Figure 2B. The TG curve shows a significant weight loss before 550 K; meanwhile, the DTG curve highlights a major decomposition peak ranging from 385 K to 550 K. The two mass-related curves are attributed to the removal of interlayer water and surface-bound hydroxyl groups in the Co/Al-LDHs structure [16,17]. With the temperature elevated, the weight loss is slightly decreased accompanied by an endothermic peak at 875 K (~600 °C) in the DSC curve, which is caused by the dehydration of the OH^- to cobalt and aluminum ions [18,19].

The SEM images (Figure 3) show significant morphological differences between SiC and R-LDHs@SCMFs as well as S-LDHs@SCMFs. For pristine SiC, smooth, angular surfaces are observed without any additional structures. In R-LDHs@SCMFs, the SiC surfaces are uniformly coated with sheet-like Co/Al-LDH microstructures. After thermal treatment, those sheet-like structures are partially melted but still maintained on the surface of the S-LDHs@SCMFs, which indicates that even if the LDHs are replaced with cobalt oxide (Co_3O_4) clusters, the sintering treatment hardly varies the morphology features between R-LDHs@SCMFs and S-LDHs@SCMFs.

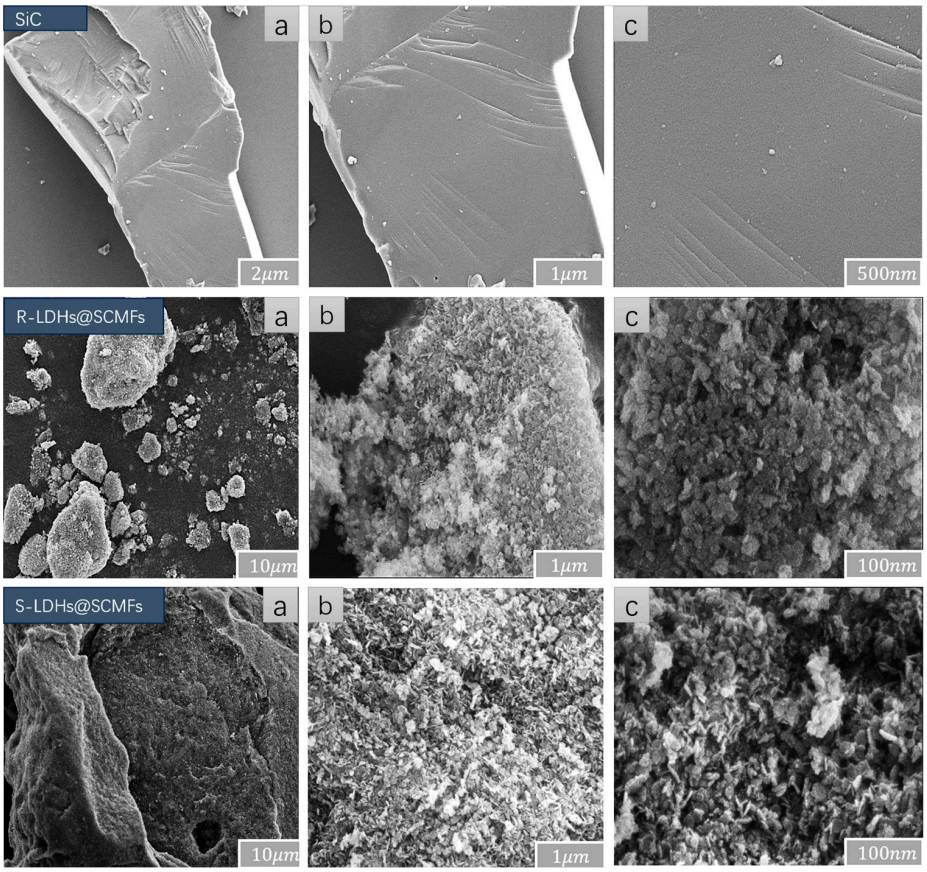

Figure 3. The SEM images of SiC, R-LDHs@SCMFs, and S-LDHs@SCMFs. Subfigures (**a**–**c**) denoted specimens were observed under various magnification times.

These morphological changes, confirmed alongside XRD and TG/DTG/DSC results, illustrate the decomposition of LDHs and the formation of Co_3O_4; the thermal treatment of the R-LDHs@SCMFs to S-LDHs@SCMFs demonstrates that controlled thermal treatment can effectively modulate the phase compositions, offering a promising strategy for improving its applicability in advanced wastewater treatment systems [2,6]. The generation of Co_3O_4 enhances the catalytic activity of SCMFs and improves reactivity for persulphate activation [10], as observed in its superior performance for aniline degradation (detailed in Section 3.2).

3.2. *Effect of Operation Conditions*

The operation conditions, including the reaction time (min), the addition of persulphate to aniline (in molecule ratio) and flow velocity (m/s) generated via backflow, are scrutinized in eliminating aniline compound.

As the addition of persulphate to aniline (denoted as P:A ratio) is constant at 8:1 and flow velocity is 0.7 m/s, the effect of reaction time (min) is surveyed in Figures 4A and 5. One may observe that approximately 16% of aniline is removed even though the reaction time is prolonged to 80 min, which indicates SiC displays weak catalytic function in activating persulphate for aniline degradation [7]. In comparison with the blank SCMFs, the LDH-coated SCMFs (R-LDHs@SCMFs and S-LDHs@SCMFs) exert superior performance in degrading aniline at all reaction times; meanwhile, the S-LDHs@SCMFs outperform the R-LDHs@SCMFs throughout the experiment. Specifically, the R-LDHs@SCMFs (or the S-LDHs@SCMFs) achieve approximately 90% (or 95%) aniline removal within 20 min. As reaction times are extended to 80 min for R-LDHs@SCMFs or 60 min for S-LDHs@SCMFs, 100% of aniline is removed. The 3D-EEM spectra (Figure 5) further corroborated the thorough elimination of aniline. One may observe that the characteristic 3D-EEM spectra of aniline solution (Figure 5A) are absent; instead, the 3D-EEM spectra of the final by-products in the effluent treated by R-LDHs@SCMFs (Figure 5B) and S-LDHs@SCMFs (Figure 5C) are emerged. Figures 4A and 5 indicate that Co/Al-LDHs plays significant catalytic role in activating persulphate, and the sintering process is further enhancing the catalytic activity and persulphate activation efficiency [13,17].

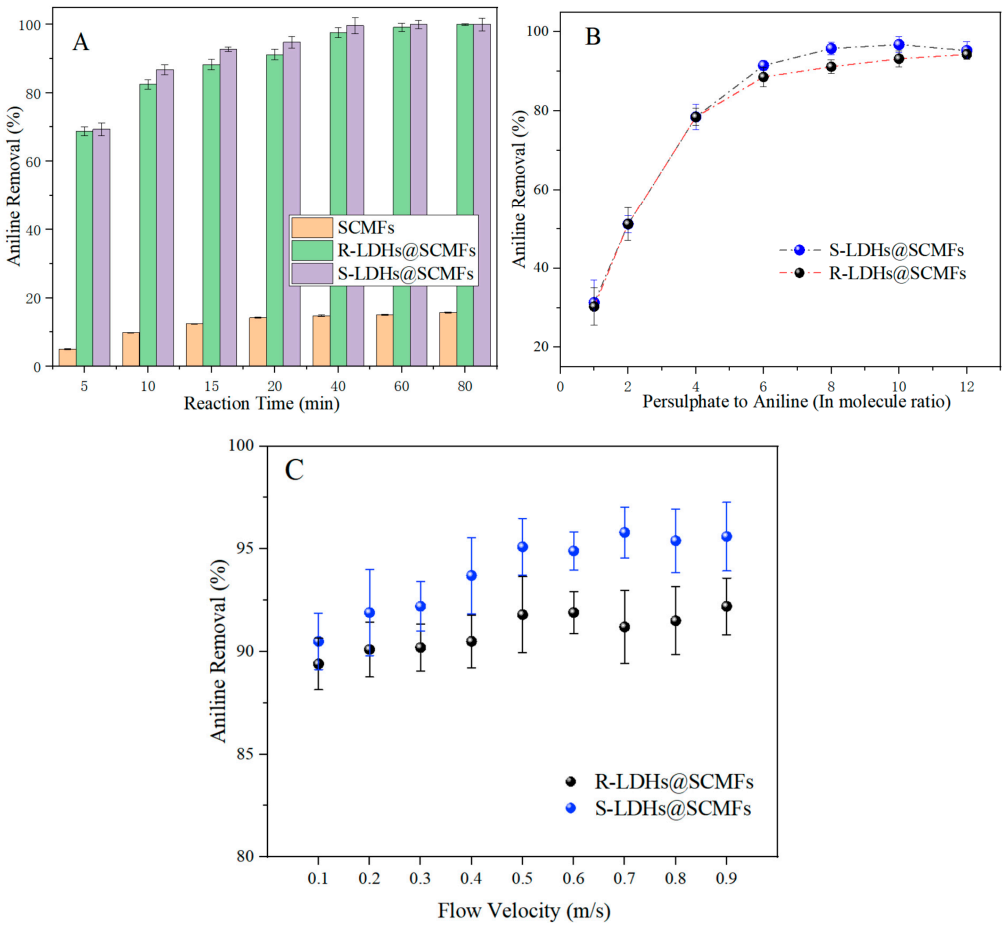

Figure 4. Effect of reaction time (**A**), addition of persulphate to aniline in molecule ratio (**B**), and flow velocity (**C**) for the removal of aniline.

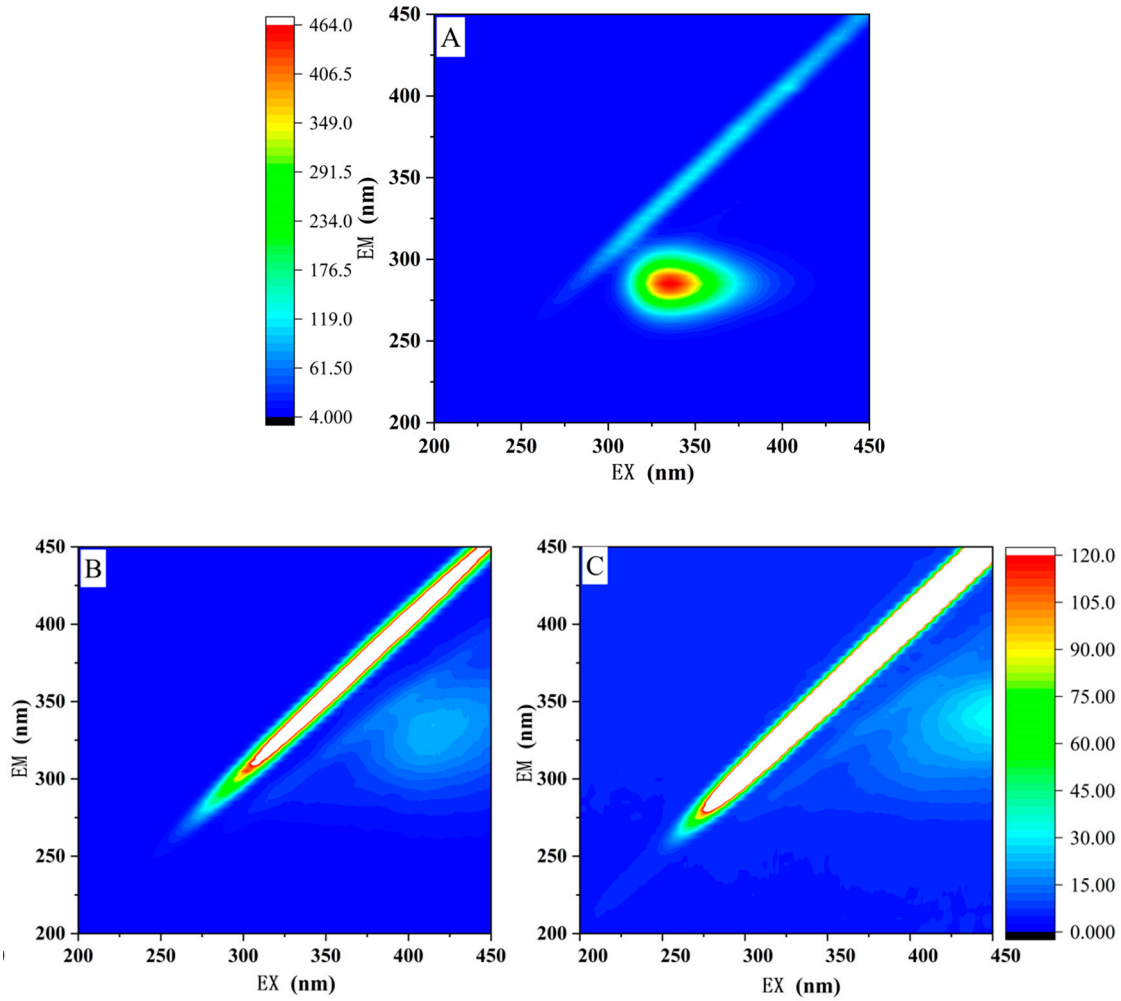

Figure 5. The 3D EEM fluorescence pattern of (**A**) the simulated aniline wastewater (100 mg/L) and the effluent of (**B**) R-LDHs@SCMFs and (**C**) S-LDHs@SCMFs, where (**B**,**C**) are both obtained under 8:1 of P:A ratio and 0.7 m/s of flow velocity, and 60 min of reaction time.

The impact of the P:A ratio on aniline removal efficiency was evaluated at a constant reaction time of 20 min and a flow velocity of 0.7 m/s, as shown in Figure 4B. One may notice both R-LDHs@SCMFs and S-LDHs@SCMFs demonstrate a consistent increase in aniline degradation efficiency as the P:A ratio rises. At a P:A ratio of 8:1, R-LDHs@SCMFs and S-LDHs@SCMFs achieve approximately 91.2% and 94.8% removal efficiencies, respectively. However, further increasing the P:A ratio beyond 8:1 leads to marginal improvements in removal efficiency. Therefore, the P:A ratio of 8:1 is adequate for achieving high removal efficiency with S-LDHs@SCMFs, balancing reagent consumption with treatment efficacy. The influence of flow velocity is shown in Figure 4C, with the reaction time and P:A ratio fixed at 20 min and 8:1, respectively. Both R-LDHs@SCMFs and S-LDHs@SCMFs exhibit increasing removal efficiency as the flow velocity rises. As the flow velocity is higher than 0.7 m/s, no significant improvement in aniline removal efficiency is obtained, indicating that optimal persulphate activation and aniline degradation are achieved at this condition [19,20].

3.3. Discrepancy of the Intermediate By-Products

Figure 4A indicates that there is a significant degradation of aniline at the early stages of the reaction (<5 min). The differences in intermediate by-products during aniline

degradation by R-LDHs@SCMFs and S-LDHs@SCMFs are analyzed via GC examination (Figure 6A,B).

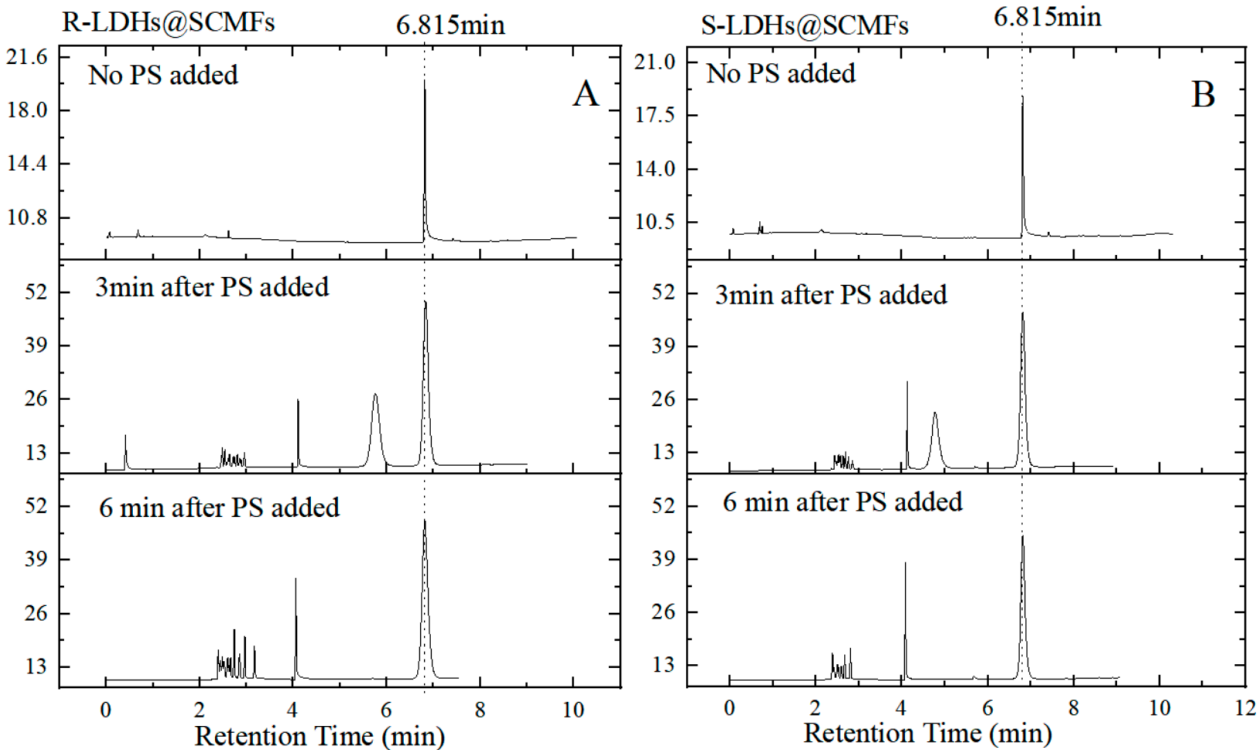

Figure 6. GC chromatography measurement (**A**) of R-LDHs@SCMFs and (**B**) S-LDHs@SCMFs operated at the early stage of reaction, where the P:A ratio and flow velocity are constant as 8:1 and 0.7 m/s; more details of the GC chromatography examination are elucidated in S4.

Without any addition of persulphate (PS), hardly discrepancy of aniline removal has emerged on R-LDHs@SCMFs nor S-LDHs@SCMFs, confirming the critical role of PS participation for the degradation of aniline. Specifically, (1) after 3 min of PS addition, discrepancies in the formation of intermediate by-products are observed at the early stages of the reaction. GC profiles reveal distinct by-products at different retention times. For R-LDHs@SCMFs, primary peaks appear at retention times of approximately 2.3 min and 3.8 min, while S-LDHs@SCMFs show additional peaks at 4.1 min and 5.6 min. (2) As the reaction time extended to 6 min, the major by-product of R-LDHs@SCMFs and S-LDHs@SCMFs are detected at similar retention time and peak intensity. This detection indicates the degradation pathways for aniline intermediates become aligned as the reaction progresses. In addition, the certification of specific reaction pathways and identification of intermediate by-products will be scrutinized in a further study. Furthermore, although the morphological features are hardly varied (Figure 3) between R-LDHs@SCMFs and S-LDHs@SCMFs, the discrepancy of the intermediate by-products implies the different reaction pathways between Co/Al-LDHs and Co_3O_4 (Figure 2A). The sintering process emerges as a key factor in enhancing the material's catalytic properties [11], leading to its superior performance across all reaction stages. Moreover, future works, including about the long-term performance stability of the reaction mechanism, will be conducted before the engineering-oriented applications.

4. Conclusions

Co/Al-LDHs were effectively used to enhance silicon carbide membrane filters (SCMFs) for activating persulphate in aniline degradation in wastewater. Under specified

conditions (20 min reaction time, 8:1 of persulphate-to-aniline molar ratio, and 0.7 m/s flow velocity), R-LDHs@SCMFs and S-LDHs@SCMFs achieved aniline degradation efficiencies of 91.2% and 94.8%, respectively. The structural and thermal analyses demonstrated that the sintering process transformed Co/Al-LDHs into cobalt oxide (Co_3O_4), significantly enhancing the catalytic activity of SCMFs. SEM imaging confirmed that the sintering process preserved the morphological integrity of the Co/Al-LDH coating. GC chromatographic analysis highlighted discrepancies in intermediate by-products. Within the first 3 min, S-LDHs@SCMFs produce a diverse array of intermediates compared to R-LDHs@SCMFs. The integration of Co/Al-LDHs coatings and sintering endows the catalytical function to the traditional SCMFs, which possess a robust solution for industrial wastewater treatment.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/w17030355/s1, Details of the examining measures and the employed instruments are given in S1: TG/DTG/DSC; S2: XRD. S3: SEM, S4: GC Chromatograph and S5: 3D Fluorescence.

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