SiC-C Composite as A Highly Stable and Easily Regenerable Photothermal Material for Practical Water Evaporation

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Solar-driven water distillation by photothermal materials is emerging as a promising way of renewable energy-driven clean water production. In designing photothermal materials, light absorption, photo-to-thermal conversion efficiency, and ability to localize thermal energy at the water-air interface are three important considerations. However, one additional consideration, regenerability, has so far slipped out of the photothermal material designs at status quo. This work reveals that there is a fouling layer formed during photothermal evaporation of real seawater (Red Sea water) and domestic wastewater, which once formed, would be difficult to remove. Herein, we synthesize a SiC-C composite monolith as an effective photothermal material where carbon acts as photothermal component and SiC serves as a heat conductor and strong structural support. The high mechanical strength of the monolithic composite makes it able to withstand repeatedly high strength physical cleaning by brush scrubbing and sonication and the anti-carbon-loss mechanism generates zero carbon loss during the physical cleaning. In the case of the domestic wastewater evaporation, the bio-

and organic foulants on the SiC-C composite monolith can be totally removed by annealing at 1000 °C in N₂ atmosphere. We believe that the SiC-C composite monoliths are promising photothermal materials in practical solar-driven water evaporation applications thanks to their highly stable and easily regenerable properties and therefore more research efforts are warranted to further improve their performances.

KEYWORDS

Solar-driven water evaporation; photothermal material; SiC-C monolith; foulant cleaning; high mechanical strength; thermal stability

INTRODUCTION

Solar-driven water distillation, which directly uses solar energy to generate water vapor and then collects condensate as fresh water, is emerging as a promising way of renewable energy-driven clean water production.¹⁻³ It is able to produce fresh water from a variety of water sources, is attractive and economical especially for small sized water plants, and can be made into point-of-use potable water production apparatus. An effective photothermal material is a key to a successful solar-driven water distillation design. Recently, many photothermal materials have been developed and showed good photo-to-thermal conversion capability, such as carbon based material,⁴⁻⁸ plasmonic particles,⁹⁻¹² organic polymers,¹³ along with many emerging new materials.¹⁴⁻¹⁶

In designing photothermal materials, high solar light absorption, high photo-to-thermal conversion efficiency, and ability to localize thermal energy at the water-air interface are three important considerations. However, one additional consideration, foulant cleaning strategy, has so far slipped out of the photothermal material design due to the fact that in the

majority of the previous works, instead of natural waters, pure water or synthetic seawater (3.5% NaCl), was used as surrogate to prove the energy efficiency of the photothermal materials.

As known, the composition of natural water is much more complex than pure water or synthetic seawater. The natural water generally contains such species as bacteria, dissolved organic and inorganic species, etc., which would lead to material fouling.¹⁷⁻²¹ Fouling is generally defined as the accumulation of unwanted compounds/substances on a material's surface, which is one of the biggest concerns in all kinds of water treatment processes and degrades the performance and shortens the service lifetime of the materials. Thus, in practical solar-driven water evaporation applications, the photothermal materials would have to face the similar fouling problems to other water treatment processes. Therefore, regenerable property should be considered in any photothermal material intended for practical solar-driven water evaporation, which unfortunately is missing in the majority of the previous photothermal materials. Envisioning typical foulants in solar-driven water evaporation, a photothermal material ought to be mechanically strong to withstand washing with flushing water, periodical physical cleaning/brushing or sonication, and potential heat treatment to remove biomass as basic foulant cleaning approaches.²²⁻²⁵

In the previous photothermal designs, composite materials are typically fabricated in which light-adsorbing materials are physically deposited onto a porous structural support, such as paper,¹¹ metal mesh,¹³ macrosporous silica,⁴ AAO membrane,²⁶ wood,⁵ etc. The necessity of the porous substrates is due to their ability to enhance light absorption by reducing light reflection and to increase the material/water interface for fast heat transfer. However, the weak interaction between the light-adsorbing materials and the structural support makes the

light-adsorbing materials easily come off the substrate during applications and also many of porous substrate supports are not mechanically strong enough, both of which would lead to these photothermal materials not able to withstand the basic foulant cleaning measures as listed above.

In searching for a porous support substrate suitable for practical photothermal applications, we were attracted by porous ceramics, which typically possess such desirable properties as high mechanical strength, excellent corrosion resistance, low cost, and desirable porous structure, etc. These attractive properties have led porous ceramics to various applications, including catalyst supports, heat exchangers, filters, among others.²⁷

Recently, porous silicon carbide (SiC) ceramics have been intensively studied due to their excellent mechanical and chemical stability, low density, high thermal shock resistance, and excellent corrosion resistance at high temperature.²⁸⁻³⁰ Very fortunately, during the preparation of SiC ceramics, carbon, a highly effective light-absorbing material, is typically coproduced as by-product, which makes the commercial products of SiC appear deep black. The carbon content in the SiC ceramics is generally uniformly distributed and more importantly is locked within the skeleton of SiC, thus representing an effective anti-carbon-particle loss mechanism.^{29, 31} Therefore, we believe that SiC and carbon composite (SiC-C) have a possibility of being a good photothermal material with a highly stable property.

Herein, we rationally design a SiC-C composite monolith as an effective photothermal material where carbon acts as the light absorber and heat converter and SiC serves as a heat conductor and strong structural support. The high mechanical strength of the monolithic composite makes it able to withstand repeatedly high strength physical cleaning by brush scrubbing and sonication and the anti-carbon-loss mechanism generates zero carbon loss

during the physical cleaning. Moreover, this work, for the first time, reveals that the natural seawater (Red Sea water) would form on the photothermal material surface the precipitation of CaCO_3 , MgCO_3 and CaSO_4 , which, once formed, are difficult to totally remove by simple water flushing. In the case of the domestic wastewater evaporation, the bio- and organic foulants on the SiC-C composite monolith can be totally removed by annealing at 1000 °C in N_2 atmosphere. We believe that the SiC-C composite monoliths are promising photothermal materials in practical solar-driven water evaporation applications thanks to their highly stable and easily regenerable properties and therefore more research efforts are warranted to further improve their performances.

EXPERIMENTAL SECTION

Chemicals and materials. Small sized silicon carbide (SiC, small particle, beta-phase, nanopowder, 45-55 nm, 70-90 $\text{m}^2 \text{g}^{-1}$, Alfa Aesar), large-sized silicon carbide (SiC, big particle, beta-phase, 99.8%, metals basis, 1 micron, 14-19 $\text{m}^2 \text{g}^{-1}$, Alfa Aesar), polycarbomethylsilane (PCMS, Sigma-Aldrich), aluminum oxide (Al_2O_3 , 40 – 50 nm, 99.5%, Alfa Aesar), yeast extract (Fluka), glutinous rice flour (AFTA brand, from Shaflout Group) and toluene (Fluka) were used in this work. Commercial synthetic seawater (ASTM D114) was from RICCA Company. All of the chemicals were purchased and used without further purification.

Fabrication of tandem-structured SiC ceramic monolith composite. SiC-C composite monolith was synthesized following a literature method, which used PCMS as a preceramic polymer and SiC mixture of two sized powers (namely, 45-55nm and 1 micron) as the

precursor. In a typical synthesis, 1.4 g large sized SiC powder (1 micron) and 0.6 g small sized SiC powder (45-55 nm) were mixed with 0.4 g PCMS in an onyx mortar and then dissolved within 20 ml toluene. It is worth mentioning that PCMS in the synthesis is both carbon precursor and also acts as cross linker.³²⁻³⁵ The gray colored paste-like mixture was evaporated on a 60°C hotplate to generate gray powdery composite. The dried composite was then ground along with 0.1 g Al₂O₃ powder in the onyx mortar, followed by sieving by 400 mesh. Al₂O₃ in this synthesis is a sintering aid, which makes possible of fabricating highly dense SiC monolith at a reduced temperature.³⁶⁻³⁷

To make a tandem structure of SiC, a sacrificial template method was used.³⁸ Different quantities of yeast (0.03 g, 0.06 g, 0.09 g and 0.12 g) and rice flour powder (0.03 g, 0.06 g, 0.09 g and 0.12 g) as the sacrificial templates were mixed with 0.45 g sieved SiC powders. The remaining sieved SiC powders were first transferred into an empty stainless steel mold (inner diameter = 4 cm) on top of which the mixture with the yeast and rice flour was then added. The mixed powders were compressed into a tablet-like monolith under a pressure of 80 MPa for 20 min. Then the compressed monolith composite was transferred into a horizontal ceramic tube furnace and subjected to a thermal treatment in N₂ atmosphere at 1200 °C for 2 hours with a temperature ramp of 2°C min⁻¹. The heat treatment removed the sacrificial template and thus generated monolithic SiC-C tandem structured composite having a porous top layer and a dense bottom layer. The tandem-structured SiC-C monoliths prepared with different quantities of templates were denoted as SiC-C (0.03y+0.03r), SiC-C (0.06y+0.06r), SiC-C (0.09y+0.09r) and SiC-C (0.12y +0.12r).

For a comparison, a single layer SiC-C composite monolith with a uniformly dense structure was synthesized in the absence of any sacrificial templates, denoted as SiC-C dense, while a

single layer SiC-C composite monolith with a uniformly porous structure, denote as SiC-C porous, was also prepared by mixing the sacrificial templates with all the sieved powders, following otherwise the same conditions.

Material characterization. Scanning electron microscopy (SEM, FEI Quanta 600) was used to characterize the morphology and powder X-ray diffraction (XRD, Bruker D8 Discover diffractometer) was used to characterize the crystal properties of the samples. The scanning of 2θ angle (degree) was made in the range of 20° to 80° with a scanning rate of $2.5^\circ/\text{min}$. Optical microscope (Olympus BX51, 5x, Olympus) was used to get the microscopic images of the surface of materials and also get the observation of the fouling layers on the surface of materials. Thermal gravimetric analysis (TGA, NETZSCH) was carried out in the range of $25\text{-}1000^\circ\text{C}$ at a heating rate of 5°C min^{-1} .

The UV/visible/infrared reflectance properties of the materials were measured using a spectrophotometer equipped with an integration sphere (UV-Vis-IR Cary 5000), with fine BaSO_4 powder as a reference baseline in the spectral range of $300\text{--}2000\text{ nm}$ at room temperature. Raman spectra were obtained on a Horiba Aramis, with a 473 nm laser wavelength excitation. Fourier transforms infrared (FTIR) spectroscopy was measured in the absorption mode within the scanning range from $650\text{ to }4000\text{ cm}^{-1}$ using an FTIR spectrometer (Thermo Scientific-Nicolet 8700). Contact angles were obtained by using an OCA 35 (Data-Physics, Filderstadt, Germany). Thermal images and temperature gradients were collected using an infrared camera (FLIR A655).

Water evaporation performance measurement. A solar simulator was used to provide the solar irradiation (Oriel Sol1A Class ABB Solar simulators, Newport) with an intensity of one sun ($\text{AM } 1.5, 1000\text{ Wm}^{-2}$). A homemade system was used in the water evaporation

performance measurements⁸. In more details, a cylindrical polystyrene (PS) container with a mouth diameter of 4.2 cm was filled with the test source water. The SiC-C composite monolith was put on top of an expanded polystyrene foam (PS) substrate, which made it able to float on the surface of water. In this work, two strips of orthometric cotton cloth were wrapped onto the SiC-C monolith together with PS substrate. The cotton cloth was immersed in the bulk water. The water container was placed on an electronic analytical scale to monitor real-time water mass loss due to evaporation. A white paper board was used as a cover around the measurement system to minimize the effect of the ambient airflow turbulence.

RESULTS AND DISCUSSION

The SiC is designed to have a monolithic tandem structure with a porous top layer and a dense bottom layer and this structure is beneficial to improve light absorbance³⁹⁻⁴⁰ and at the same time to give the structure a strong mechanical property and thus ability to withstand strong physical forces during physical cleaning (Figure 1).

In the synthesis of SiC-C monolith in this work, Al_2O_3 is used as a sintering aid, which effectively lowers the calcination temperature to 1200 °C from otherwise 1500 °C without the sintering aid. PCMS is the carbon source in the synthesis and it also acts as cross linkers, which help the cross-linking and thus the generation of compact SiC monolith. The synthesis procedure of the tandem-structured SiC-C composite monolith is presented in Figure S1.

Figure 1 The conceptual diagram showing the design of SiC-C composite monolith, its fouling during practical water evaporation and the foulant cleaning approaches.

Figure 2 (1) digital photos of SiC-C (0.09y + 0.09r) monolith showing its diameter and thickness. (b) SEM images of cross-section of SiC-C (0.09y + 0.09r) monolith. Inset in Figure 2b is the contact angle of the surface of SiC-C

(0.09y + 0.09r) monolith ($\sim 10^\circ$). (c) and (d) the zoomed-in images of the bottom dense layer of SiC-C (0.09y + 0.09r) monolith.

The samples prepared with different amount of two kinds of sacrificial templates (Figure S2 and S3) are presented and discussed in Supporting Information (SI) (Figure S4, S5, S6 and S7). Two different sizes of SiC precursor powders are used in order to get a compact structure and the sizes of the SiC precursor powders are presented in Figure S8. All the samples have the same diameter of ~ 3.93 cm and thickness of ~ 1.1 mm (Figure 2a), which appears visually deep black. From the cross-sectional SEM image of SiC-C (0.09y+0.09r) (Figure 2b), a clear boundary between the top porous layer (~ 300 μm thickness) and the bottom dense layer (~ 800 μm thickness) can be seen. The porous top layer expectedly consists of macro-pores (1-70 μm) while the bottom dense layer contains only nano-pores (50-400 nm). As comparisons, single layered dense (Figure S5a) and porous (Figure S5f) SiC-C monoliths are prepared, showing relatively uniform pores thorough the entire monoliths. Inset in Figure 2b shows that the surface of SiC-C monolith is hydrophilic with a contact angle of 10° .

Figure 3 (a) TGA curve of SiC-C (0.09y + 0.09r) monolith in air atmosphere in the temperature range of 25-1000 $^\circ\text{C}$, with a heating rate of 5 $^\circ\text{C}/\text{min}$. (b) Raman spectra of SiC-C (0.09y + 0.09r) monolith measured with 473 nm laser wavelength excitation, obtained from the top surface and the cross sectional surface.

202 The crystalline phases present in the raw SiC powders as well as in the sintered ceramic SiC-
203 C composite monolith are identified by X-ray powder diffraction (XRD) in Figure S9. XRD
204 spectra of SiC precursor powders and the monolithic samples annealed at 1200 °C show that
205 the SiC-C monolith samples are single phase, having the crystal structure of β -SiC (JCPDS
206 74-2307). No peaks related to the isolated hexagonal SiC polytypes are observed.

207 The amount of free carbon content, which is photothermal component, of SiC-C monolith is
208 investigated through thermal air-oxidation behavior of the residue obtained from TGA curve
209 (Figure 3a). The TGA curve in the temperature range of 25-1000 °C shows the succession of
210 two steps. The initial weight loss (4.6%) occurring in the 310-776 °C temperature range is
211 assigned to the air oxidation of the free carbon, which indicates the content of free carbon in
212 SiC-C (0.09y +0.09r) monolith is sufficiently high and around 4.6%. The weight increase
213 above 776°C is due to air-oxidation of SiC into SiO₂ as determined by XRD (Figure S10). The
214 amount of free carbon in our case is a good compromise between light absorption and
215 material's hardness. The free carbon content in the synthesis of SiC-C composite can be
216 potentially adjusted, but it can drastically affect the hardness and mechanical strength of SiC-C
217 monolith far beyond a simple effect of porosity.³¹ The carbon content is not adjusted in this
218 work given the good balance of the light absorption and mechanical robustness of our SiC-C
219 monolith.

220 Carbon structure is determined by Raman spectroscopy with a 473 nm laser wavelength
221 excitation. The set of two large bands located at 1358 cm⁻¹ (D1 band) and 1593 cm⁻¹ (G band)
222 is typically encountered for an amorphous structure of carbon. Raman spectra obtained from
223 the top surface and the cross-section of the monolith show the similar typical bands from C-C,
224 meaning that the free carbon in tandem-structured SiC-C monolith distributes quite uniformly.

For comparisons, Raman spectra of SiC precursor powders are also presented in Figure S11. Regarding the chemical stability, a sample of SiC-C monolith is immersed in sulfuric acid (0.2%), nitric acid (0.3%) and NaOH (0.4%), respectively.⁴¹ Judging from the photos taken before and after the chemical treatment (Figure S12), SiC-C corrosion is observed after NaOH treatment, however, there was not obviously visual difference in the appearance of the sample after acid treatment, which shows its stability in acid and is in agreement with the literature results.⁴²⁻⁴³

Figure 4 Diffusion reflectance measurements of SiC-C monoliths in UV-Vis range (a) and in IR range (b). (c) Time-course of the temperature of SiC-C (0.09y + 0.09r) monolith and SiC-C dense monolith. Inset images are the steady-state temperature under solar illumination (AM 1.5) of SiC-C (0.09y + 0.09r) monolith and SiC-C dense monolith respectively.

The UV-Vis-IR reflectance measurement results show that the SiC-C monoliths have a relatively low light diffusion reflection in both UV-Vis spectra (Figure 5a) and IR spectrum (Figure 5b), indicating that the SiC-C monoliths have a good light absorption within almost the entire spectral range of solar irradiation, i.e., from ~300 nm to ~ 2.0 μm . As seen from Figure 4a, the tandem structure shows less diffusion reflection from 300 to 800nm than both SiC-C dense monolith and SiC-C porous monolith, indicating the role of the rationally

designed tandem structure on the light adsorption enhancement. As can be seen, the diffusion reflection is decreasing as the increasing amounts of the sacrificial templates, which is consistent with the visual color changes of the samples (Figure S2). On the other hand, the difference in the IR absorption by the structural design is not as significant (Figure 4b)

Having confirmed the light absorption enhancement of the SiC-C monoliths, their photo-thermal properties are investigated. Figure 4c presents the materials' surface temperature in air as a function of illumination time under solar simulation of one sun. Upon light illumination, the surface of SiC-C monolith rises quickly to a steady-state temperature after around 700 seconds. Inset in Figure 4c is IR images of the SiC-C monolith and SiC-C dense monolith obtained after illumination for 900 s. The IR images of other samples after illumination for 60 s and 900 s are presented in Figure S13 and Figure S14. The SiC-C dense monolith has a lower surface temperature of $\sim 60.1^{\circ}\text{C}$ compared to the SiC-C (0.09y+0.09r) monolith of $\sim 68.3^{\circ}\text{C}$, which indicates that the tandem-structured SiC-C monolith has a better photo-to-thermal conversion performance and is partially due to the tandem monolith having higher light absorption. Upon light off, both materials have a quick drop in their surface temperatures in response.

Figure 5 Time-course of (a) DI water and (c) 3.5% NaCl solution evaporation performance by SiC-C monoliths under various conditions. The average (b) DI water and (d) 3.5% NaCl solution evaporation rate of final 30 minutes.

Figure 5a and 5b present the time course of DI water evaporation by SiC-C monoliths with different amount of sacrificial templates under solar irradiation. As can be seen, the amount of water evaporated increases with the increase of the sacrificial templates in the preparation process. All of the tandem-structured SiC-C monoliths evaporate more water than SiC-C dense monolith. Evaporation of 3.5% NaCl solution, a simplified surrogate of seawater, is tested and presented in Figure 5c and 5d. The solar-to-evaporation efficiency η of DI water and 3.5% NaCl solution evaporation are calculated by following literature method (SI) and Figure S15 and Figure S16 present η as a function of illumination time. Generally, SiC-C monoliths all show satisfactory energy efficiency. For example, the η of SiC-C (0.09y+0.09r) is $\sim 71\%$ for DI water and is $\sim 65\%$ for 3.5% NaCl solution, which make them promising for practical applications.

Figure 6 XRD spectra of SiC-C (0.09y+0.09r) monolith with salts precipitation from 3.5% NaCl solution (a) and after water flushing cleaning (b); salts precipitation from commercial synthetic seawater (c) and after water flushing cleaning (d). Insets are microscopic images of top view of each sample. (^ indicates SiC, * indicates NaCl, ~ indicates MgCO₃, @ indicates CaCO₃ and # indicates CaSO₄)

There is clearly a layer of solid precipitates on the surface of the SiC-C monolith after 3.5% NaCl solution evaporation for 3 hours (Figure S17a), which is further confirmed by microscopic images (Figure 6a-2). The XRD spectra (Figure 6a-1) determine that the salt crystals are only NaCl. Simple flowing water flushing is applied to clean the surface salt off the material. It turns out that the surface salts can be easily removed this way (Video 1). The SiC-C monolith after the water flushing shows a visually clear surface (Figure S17b). The XRD spectra (Figure 6b-1) and microscopic image (Figure 6b-2) further confirm that all the peaks from NaCl disappear after the cleaning. The results of easy NaCl removal are consistent with the literature reports.⁴⁴ Later on, water evaporation test was conducted by using commercial synthetic seawater whose ion composition is shown in Table S1. The SiC-C

monoliths after the commercial synthetic seawater evaporation were further investigated by XRD who results show that the salt crystals on the SiC-C surface were NaCl, MgCO_3 , CaCO_3 and CaSO_4 (Figure 6c-1). After water flushing cleaning, highly soluble salt precipitate, such as NaCl, was removed, suggesting that the less soluble salts on the surface of material are potential scaling species.

Figure 7 Water evaporation performance of Red Sea water by the SiC-C (0.09y+0.09r) monolith. (a) Water evaporation rate as a function of evaporation time lasting for 6 cycles (c-1 to c-6). In each cycle, light is on for 3 hours and light is off for 1 hour. (b) Water evaporation rate as a function of evaporation time for 7 cycles, with water flushing and brush scrubbing at the end of each cycle of the first 6 to clean surface foulants and with additional sonication before the 7th cycle.

The natural Red Sea water is then sampled and used for the water evaporation test under the otherwise same conditions and different results are observed. In order to investigate the effect of the fouling layers on the water evaporation performance over long-term operation, cycle stability is investigated and the water evaporation performance is tested for 6 cycles with each cycle consisting of a light-on step for 3 hours and a light-off step for 1 hour.

Figure 7a presents the water evaporation rate as a function of evaporation time for 6 cycles without any surface foulant cleaning. The water evaporation rate decreases from $1.05 \text{ kg m}^{-2} \text{ h}^{-1}$ in the first cycle to $0.82 \text{ kg m}^{-2} \text{ h}^{-1}$ in the sixth cycle, representing a 21.9% performance decline. Figure S18a presents digital photos of the material surface after each cycle and clearly show thick fouling layers on the surface of the SiC-C (0.09y+0.09r) monolith. The presence of the fouling layers and more importantly significantly reduced performance warrants certain foulant cleaning methods to be taken on the materials. In this case, water flushing and brush-scrubbing are taken to clean the foulants off the materials (Figure S18b-1 to b-4, Video 2 and Video 3).

Figure 7b presents the water evaporation rates as a function of evaporation time for 6 cycles, with the foulant cleaning via water flushing and brushing after each cycle. The water evaporation rate decreases in this case from $1.06 \text{ kg m}^{-2} \text{ h}^{-1}$ in the first cycle to $0.96 \text{ kg m}^{-2} \text{ h}^{-1}$ in the sixth cycle, showing a much less 9.4% decline compared with the 21.9% decline in the case without any foulant cleaning. Figure S19 presents the microscopic images of Si-C monolith with fouling layer, after cleaning with water flushing and after cleaning with brushing. The obvious difference between the two cases indicates the importance of periodical physical cleaning of the photothermal material in order to keep a relatively stable performance.

Then the same SiC-C monolith is further cleaned by sonication in DI water for 30 min (Video 4), and the evaporation performance is presented in Figure 7b cycle-7, where the evaporation rate is recovered to 94.3% of the evaporation rate in first cycle. It is worth emphasizing that the sonication water container does not show any significant color change before and after sonication the SiC-C monolith in it (Figure S18c-1 and c-2), implying there are not carbon particles coming out of the SiC-C monolith and into the water. The anti-carbon-loss

mechanism in the SiC-C monolith effectively locks the photothermal carbon into the matrix of SiC securely. It has to be pointed out that most of the previous photothermal materials in the literature cannot withstand water flushing or sonication treatment for an extended time without losing their structural integrity or losing their photothermal components (Figure S20).⁴⁵ More tests have been done to investigate the mechanical property of SiC-C monolith by a simple pressure test (Figure S21), whose results show that a SiC-C (0.09y+0.09r) monolith sample (with diameter of 39.3 mm and thickness of 1.1 mm) can hold a relative high pressure as high as 0.84×10^6 Pa.

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Figure 8 Top view of digital photos and microscopic pictures of SiC-C (0.09y+0.09r) composite monolith under different conditions in Red Sea water for water evaporation: the freshly prepared SiC-C (0.09y+0.09r) monolith (a-1 and a-2), the SiC-C (0.09y+0.09r) monolith with fouling layer after water evaporation for 6 cycles (b-1 and b-2), the fouling layers after cleaning with water flushing (c-1 and c-2), after cleaning by brush scrubbing (d-1 and d-2), and after cleaning with sonication for 30 min (e-1 and e-2). a3-e3 present the XRD spectra of each sample. (^ indicates SiC, * indicates NaCl, ~ indicates MgCO₃, @ indicates CaCO₃, # indicates CaSO₄, and ? indicates SiO₂ (Cristobalite)).

To ascertain the fouling species and their removal during the above experiments, more detailed surface morphologies and XRD based speciation analysis are conducted. Figure 8 presents the digital photos and top view microscopic pictures of SiC-C (0.09y+0.09r) monolith under different conditions: monolith freshly prepared (Figure 8a), monolith with fouling layer after water evaporation with Red Sea water for 6 cycles (Figure 8b), monolith after cleaning by flushing DI water (Figure 8c), monolith after cleaning with brush scrubbing (Figure 8d), and monolith after cleaning with sonication for 30 min (Figure 8e). From Figure 8b-2, there is a severe surface fouling of SiC-C (0.09y+0.09r) monolith after 6 cycles of Red Sea water evaporation. The XRD analysis reveals that the fouling species are crystals of NaCl, MgCO₃, CaCO₃ and CaSO₄ (Figure 8b-3), which is in agreement with the ion composition of Red Sea water (Table S2). A peak from SiO₂ (Cristobalite) was found interestingly at around 22°, which is different from the commercial seawater. Simple flowing water flushing, the same as the previous 3.5% NaCl solution case, is applied to clean the material (Video 2) and after which there are still a lot of solid residues left on the surface of the monolith (Figure 8c-1 and c-2). However, the XRD results show that the simple water flushing successfully removes NaCl out of the material as the peaks from crystals of NaCl disappears entirely after the flushing (Figure 8c-3). Apparently, these leftover fouling species after the simple flushing are these carbonate and sulfate species, which cannot be removed from the materials by the simple water flushing probably due to their low water solubility.

Strong brush scrubbing is applied to clean the material surface under flowing tap water (Video 3), after which the peaks from MgCO₃, CaCO₃ and CaSO₄ are reduced significantly (Figure 8d-3) and the material surface looks much cleaner (Figure 8d-1). However, this step still does not remove these sulfate species completely as shown in XRD pattern as well as in

Figure 8d-2. In order to further clean the SiC-C monolith, the sample is sonicated while immersed in DI water for 30 min (Video 4). After the sonication, there are not any visible residues on the surface of the SiC-C monolith (Figure 8e-1 and 8e-2). As matter of fact, the sonication successful removes all inorganic fouling species, leaving behind only peaks for SiC monolith (Figure 8e-3) and meaning that the SiC-C monolith is completely regenerated.

First, our results clearly demonstrate that fouling will be a very serious issue for the practical solar-driven water evaporation applications where natural and typically dirty water is involved. Thus having regenerable property is very much needed for the photothermal designs intended for these applications. Secondly, it is the first time that the difference in fouling behaviors of solar-driven evaporation of the natural seawater (Red Sea water) and 3.5% NaCl solution is reported. While surface NaCl salt precipitate on the photothermal material can be easily removed by simple water flushing, the carbonate and sulfate species of MgCO_3 , CaCO_3 , CaSO_4 , etc. are difficult to remove once they precipitate on the surface of photothermal materials. For example, the solubility of CaSO_4 increases with increasing NaCl concentration, which explains the fact that there is dissolved CaSO_4 in natural seawater and also the fact that once it is precipitated it would be difficult to remove by simple water flushing given its small water solubility in water.⁴⁶ The carbonate species, such as CaCO_3 , whose solubility decreases with increasing temperature, is one of the main species for scaling.⁴⁷⁻⁴⁸ The term fouling is used in the manuscript indiscriminately thereafter to describe the problem caused by salt scaling and organic species induced fouling.

Most of the previous photothermal works investigating seawater evaporation oversimplify their experimental conditions by using 3.5% NaCl solution. The fouling scenarios of 3.5% NaCl solution and natural seawater can be drastically different. The scaling potential can also

388 be confirmed by calculation of Langelier Saturation Index (LSI), which indicates the driving
389 force for scale formation depending on factors such as water pH, the saturation pH (pH_s),
390 temperature, salinity, calcium hardness and alkalinity of water.⁴⁹ The details of LSI
391 calculation can be found in supporting information. The calculated LSI is 0.38, which means
392 that the tested seawater from Red Sea is supersaturated with respect to calcium carbonate and
393 there is a high potential for scaling occurring. As for the solar-driven water evaporation
394 system, the concentration of salts on the surface of photothermal material is increasing
395 monotonically during the process of water evaporation. In addition, LSI is temperature
396 sensitive, which means LSI becomes more positive as the water temperature increases. In
397 other words, solar-driven water evaporation by photothermal material may intensify the water
398 supersaturating condition and thus have serious scaling concern. For the natural seawater
399 solar-driven evaporation, the photothermal material design, in addition to light absorption,
400 heat conversion, and heat concentration requirements, ought to have another requirement of
401 mechanical strength and stability so the material can survive envisioned fouling control
402 measures, including cascading means of water flushing, surface brushing, and sonication to
403 have inorganic species in fouling layer removed. In this regard, the SiC-C monolith in this
404 work provides a good example of a sturdy and effective photothermal materials towards
405 durable real world application of solar-driven water evaporation.

Figure 9 Top view digital photos and microscopic pictures of SiC-C (0.09y+0.09r) monolith under different conditions with domestic wastewater (treated after screen, collected from WWTP at KAUST): (a-1 and a-2) the freshly prepared SiC-C (0.09y+0.09r) monolith, (b-1 and b-2) the SiC-C (0.09y+0.09r) monolith with fouling layers after water evaporation in wastewater and (c-1 and c-2) the SiC-C monolith with fouling layers after cleaning with water flushing and brushing. a2-c2 present the microscopic images of each sample (5X). a3-c3 present the XRD spectra of each sample. (^ indicates SiC, X indicates unnoted species in wastewater). Curve in a-4, b-4 and c-4 present the FTIR results of each sample under different condition: freshly prepared SiC-C (0.09y+0.09r) monolith (a-4), with fouling layer from wastewater evaporation (b-4) and with fouling layer from wastewater evaporation after annealing treatment at 1000 °C in N₂ (c-4).

Furthermore, the SiC-C monolith is challenged by solar-driven domestic wastewater evaporation. The domestic wastewater sample right after physical screening was collected from WWTP at KAUST, Saudi Arabia. In addition, the water quality test of each water sample is presented in Table S3 in SI. As expected, a thick fouling layer is observed on the

surface of the SiC-C monolith after wastewater evaporation and the XRD analysis reveals some unknown species on the material surface (Figure 9b).

For the fouling layer coming from the domestic wastewater, simple flowing water flushing is ineffective (Figure 9c-1 and 9c-3, Video 5). The main fouling species in this case are organic substances as indicated by FTIR spectrum (Figure 9b-4). The FTIR spectra show the dominant functional groups in the fouling layer are amides with absorbance peaks at 3319, 1629, 1541 and 1412 cm^{-1} .⁵⁰ The peak at 2934 cm^{-1} indicates the C-H bond of alkyl. The peak observed at 1238 cm^{-1} could be due to the presence of $-\text{CH}_3$ and C-H bond in methyl group.⁵¹ A sharp peak at 1020-1070 cm^{-1} exhibits the character of carbohydrates or carbohydrates-like substances.⁵² A freshly fouled SiC-C monolith after the wastewater evaporation is annealed at 1000 °C in N_2 atmosphere and Figure 9c-4 shows a photo of the fouled SiC-C monolith after annealing treatment. On the one hand, the decrease in the number of FTIR peaks is observed, which means that annealing treatment successfully mineralizes most of the organic compounds in the fouling layer.⁵³ On the other hand, the SiC-C monolith preserves compositional and structural integrity due to its excellent thermal stability.

CONCLUSIONS

This work draws attention onto the fouling issue during practical applications of solar-driven water evaporation of real seawater and wastewater. At the same time, this work demonstrates that the tandem-structured SiC-C monolith, consisting of a top porous layer and a bottom dense layer, is a promising photothermal material towards practical applications. The material has satisfactory solar-to-evaporation performance and more importantly high mechanical

strength and durability. These properties make the material able to withstand essential fouling control approaches, water flushing, brush scrubbing and sonication, which none of the previous photothermal materials is able to. Moreover, the high thermal stability of the material allows for 1000°C heat treatment to remove organic foulants. This study shines light on the potential use of ceramic-based materials as photothermal materials in practical solar-driven seawater desalination and wastewater treatment.

ASSOCIATED CONTENT

Supporting Information. This material is available free of charge via the Internet at <http://pubs.acs.org>.

Schematic image of synthesis process of SiC-C monolith; digital photos, SEM and microscopic images of precursors, SiC-C monolith samples and fouled surface of SiC-C samples; XRD for SiC-C monolith samples and precursors under different conditions; Raman for SiC precursors; digital photos for chemical stability test; IR images for SiC-C monolith samples under illumination for 60s and 900s; equations for water evaporation rate and solar to evaporation efficiency calculation; the average evaporation efficiency for DI water and 3.5% NaCl solution under different conditions; digital photos of SiC-C monolith with salts precipitation after 3.5% NaCl solution and commercial synthetic seawater evaporation; digital photos of SiC-C monolith during evaporation process and cleaning process; comparison of mechanical stability with other materials; digital photos showing pressure test process; table of ion composition of the commercial synthetic seawater, the sample of Red Sea water and water quality of different water samples; calculation of Langelier Saturation Index (LSI).

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TOC

A highly stable and easily regenerable SiC-C composite as an efficient photothermal material is designed for practical solar-driven water evaporation.