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Treating graywater using quartz sand filters: the effect of particle size, substrate combinations, and reflux ratio

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Abstract

To achieve simple, efficient, and low-cost treatment and reuse of graywater, this study evaluated the effect of particle size, substrate combination, and reflux ratio on the treatment of graywater by quartz sand filters. Three 1,500 mm column filters were fed with synthetic graywater at a hydraulic loading rate of 1.87 $m^3/(m^2$ d) for approximately 7 months, and the particle sizes, combinations, and reflux ratio were set according to a layered plot design. Results showed that the purification performance followed the order combination > reflux ratio > size. The best combination of particle size was 4–8 mm and 1–2 mm quartz sand. It removed up to 90%, 93%, and 36% of turbidity, biochemical oxygen demand, and methylene blue active substances (MBAS), respectively. In addition, scanning electron microscopy showed that differences of the microforms and structures for quartz sand particles of different sizes, and the attachment of substrates caused a similar change in the microstructure of the quartz sand surface. This explained, to some extent, why there was no significant difference in the experimental results for each single stage. According to these results, graywater purified using a quartz sand filter is suitable for uses such as irrigation and flushing toilets.

Keywords: Graywater; Particle size; Quartz sand filter; Substrate combination; Reflux ratio

1. Introduction

With the increase in the population growth rate and climate and industrial development, a notable amount of wastewater is produced in both arid zones and very dense urban areas [1]. The balance between freshwater decrease and wastewater treatment from various resources is essential for the sustainability of water resources [2]. Currently, graywater is regarded as an alternative resource for water used for non-drinking purposes instead of expensive and risky measures such as rainwater collection by constructing reservoirs and dams and exploitation of groundwater and seawater desalination [3]. Graywater is wastewater originated from household bathrooms, dishwashers, and laundries. It accounts for approximately 50%–80% of total domestic sewage [4]. The remaining part is blackwater,

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which refers to sewage discharge from toilets. Furthermore, graywater recycling and reuse can provide reclaimed water in sufficient amounts to flush toilets, wash cars, and water gardens; this can reduce household potable water consumption by 29%–47% [5]. To achieve sustainable development goals, graywater must be treated according to a specified standard before being discharged into water bodies such as oceans, rivers, and lakes.

According to previous studies [4,6], graywater has lower concentrations of contaminants such as pathogens, nitrogen, and phosphorus, and a lower chemical oxygen demand (COD) level, than blackwater. This means it is more easily treated to meet discharge standards; the only exception is anionic surfactants. The resulting increased focus on the treatment and reuse of highly variable real graywater led to some researchers to create graywater with stable properties artificially [7]. Furthermore, growth in the recycling and reuse of graywater enables applications in a wide range of physical [8], chemical [9], and biological [10] treatment techniques. Membrane bio-reactors were feasible and efficient to treat graywater. However, membrane fouling is a problem that cannot be ignored [10,11]. Physical-chemical treatment techniques have a suitable effect using electricity, high pressures, or agents, but the treatment volume is small and the cost is high [12,13]. Constructed wetland for graywater treatment is stable, effective, and reliable, but it covers a large area and it is prone to breed mosquitos [14,15]. Therefore, it is necessary to find novel approaches such that on-site graywater recycling technologies become safe, technically practical, and economically viable. One of the better treatment techniques is a quartz sand filter [16].

Quartz sand is a silicate mineral widely used for glass, architecture, chemicals, and petroleum industries owing to its outstanding ability to resist wear and its chemical stability. It is also widely used as a technical and economical medium to filter domestic wastewater [17] and river water [18] and to treat oily wastewater [19]. Quartz sand filters cover a relatively smaller area than constructed wetlands [6] and have lower operational and management costs than a membrane bio-reactor [10] and nanofiltration [20]. Furthermore, quartz sand is a widely available material with a rough and porous surface that can absorb many contaminants [21]. The effects of the reflux ratio for wastewater were also investigated in previous studies [22,23].

For low-cost, efficient, and simple operation and management of quartz sand filters, this study mainly aimed to investigate the effects of particles size, combinations of particles size, and reflux on graywater treatments using a quartz sand filter. Based on various experiments, we determined the best scheme of graywater treatment in quartz sand filters and evaluated the possibility of reusing treated graywater for flushing toilets or irrigation.

2. Materials and methods

2.1. Synthetic graywater

To maintain the consistency of the treatment, synthetic graywater was used as an influent in the quartz sand filter. The chemical composition of the graywater [7,24] is given in Table 1, and all agents were analytical reagents.

Table 1
Chemical composition and concentrations of synthetic graywater

Chemical composition	Per litre of water
Lactic acid	41.7 µL
Cellulose	50 mg
Sodium dodecyl benzene sulfonate (SDBS)	25 mg
Glycerol	79.1 µL
Sodium hydrogen carbonate	35 mg
Sodium sulfate	25 mg
Septic tank effluent	10 mg

The water quality parameters of the synthetic graywater were pH, dissolved oxygen (DO); turbidity; biochemical oxygen demand (BOD_5); COD; ammonia nitrogen (NH_4^+–N), total nitrogen (TN), and total phosphorus (TP) levels; and methylene blue active substances (MBAS). The anionic surfactants present in the water quality index were detected by testing the MBAS of samples in this study. Sodium dodecyl benzene sulfonate (SDBS), which is an anionic surfactant, is the main component of various detergents. Moreover, cellulose was used to simulate suspended solid in graywater. Nitrogen, phosphorus, and microorganisms came from a septic tank effluent. Therefore, the synthetic graywater had the same quality parameters of real graywater. During the realization of the experiments, the solution was daily made and septic effluent stored at 4°C was taken from the septic tank once a month.

2.2. Experimental setup

The filter material was quartz sand from Dongyuan, Guangdong province, China. Note that quartz sand is commonly used to purify water [21]. Its physical and chemical properties are shown in Table 2. The height of the filler zone was 700 mm in the column.

The bottom of the column was a 250 mm layer of coarse gravel from Hangzhou, Zhejiang province, China. The 1,500 mm filter column was made of PVC pipe with a diameter of 150 mm. The top of the column had a sprinkler. The flow of the peristaltic pump (Kamoer, Shanghai) can reach 352 mL/min. The setup for the quartz sand filter is shown in Fig. 1.

Experiments were conducted for 7 months and each stage took approximately 2 months. The particle size, substrate combinations, and reflux ratio were successively studied in three stages. In the first stage, grains of quartz sand with three different sizes, namely small (0.5–1 mm (S)), medium (1–2 mm (M)), and large (4–8 mm (L)), were filled in three separate filter columns. In the second stage, the quartz sand was divided into three substrate combinations: medium- and small-sized quartz sand (MS), large- and medium-sized quartz sand (LM), and large- and small-sized quartz sand (LS). These combinations were placed on the upper and lower halves of the three-filler zone, respectively. Finally, based on the second Finally, based on the second stage, a reflux ratio of 1:2 was established in the medium- and small-sized quartz sand filter (RMS), large- and medium-sized

Table 2
Characteristics of the quartz sand sample

Chemical composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	Cr_2O_3	Loss on ignition
Value (%)	99.72	0.066	0.004	0.01	0.0025	0.012	0.007	0.01	0.00014	0.13

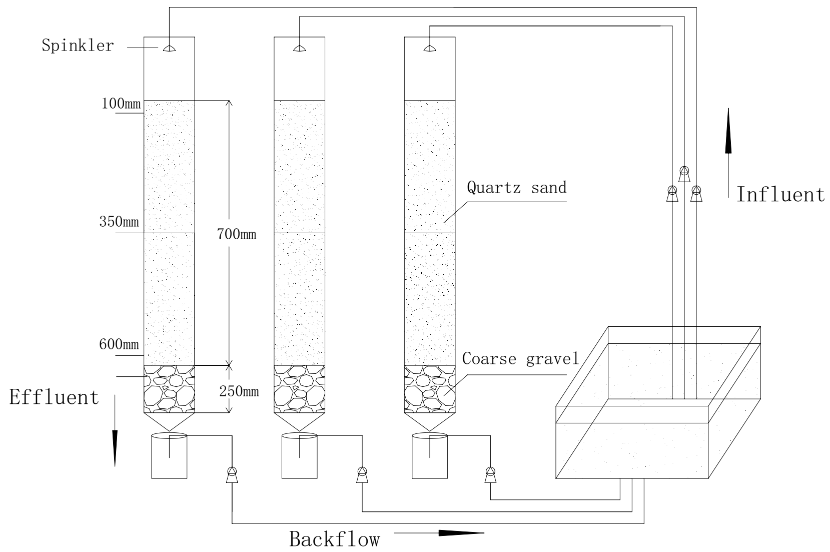

Fig. 1. Setup of quartz sand filter.

quartz sand filter (RLM), and large- and small-sized quartz sand filter (RLS) and the flow of reflux pump was set at 22.5 mL/min. During the realization of the experiments, the influent rate for each peristaltic pump was set at 45 mL/min at a hydraulic loading rate of 1.87 $m^3/(m^2 d)$; the filtration velocity of non-pressurized sand filter was 0.08 m/h, and an interval water of 15 min was adopted. The backflow was mixed with raw water and then filtered by quartz sand filter.

2.3. Sampling and analyzing graywater

Samples of the inflow and outflow from the different filters were collected every 3 d, after the operation of each stage became stable. The samples were analyzed for pH, turbidity, DO, COD, and BOD_5 by a HQ40d portable multi-parameter meter, a 2100Q portable turbidimeter, an HQ30d fleximeter, a Hach Lange DR 2800 spectrophotometer, and a BODTraK™II meter (Hach, Loveland, CO, USA), respectively. The MBAS were also measured with a Hach Lange DR 2800 spectrophotometer. The NH_4^+–N concentration was measured using the Nessler's reagent colorimetric method; the TN level was determined using the alkaline-potassium-persulfate-digestion ultraviolet spectrophotometric method; and the TP was determined using the ammonium-molybdate spectrophotometer method. Furthermore, quartz sand samples of three sizes were scanned with a Nova200 NanosSEM (FEI, Hillsboro, OR, USA) before the experiment. After the second stage, samples in the upper 100 mm (MS-u), middle 350 mm (LM-m), and lower 600 mm (LS-l) parts of each filling zone were analysed using scanning electron microscopy (SEM). Therefore, 12 samples were analysed by SEM. The data were examined for normality and homogeneity of the variations using *T*-test in SPSS software.

3. Results and discussion

3.1. pH

The pH range for all of the inflows (6.58 ± 0.60) was similar to that of real graywater from separated sources, for example, bathroom discharge, and from mixed sources [25]. The quartz sand filter had a negligible effect on the pH [26]. During the filtration process, the outflow pH was affected by the concentration of free hydrogen ions in the filtrate media [8]. Moreover, quartz sand has a rough and porous surface that facilitates ion exchange. This type of surface has a negligible effect on pH in this experiment [17,19]. Table 3 shows the significance analysis for a variety of conditions. There were no statistically significant differences ($p > 0.05$ throughout this experiment) between the pH values of the outflow for the different filters in the three stages.

3.2. Dissolved oxygen

Fig. 2a shows the quality of the synthetic graywater in the inflow and overall outflow. The DO value in the freshly prepared synthetic graywater was approximately 9.0 mg/L. The outflow DO values were stable for differently sized quartz sand particles, which performed better independently rather than in combination and reflux in terms of reducing

Table 3
Significance of the effects of three stages

Parameter	pH	Turbidity	DO	COD	BOD_5	NH_4^+–N	TN	TP	MBAS
S•M	n.s	n.s	n.s	s	n.s	n.s	n.s	n.s	n.s
S•L	n.s	s	n.s	s	n.s	n.s	s	n.s	n.s
M•L	n.s	s	n.s	s	n.s	n.s	s	n.s	n.s
MS•LM	n.s	n.s	n.s	n.s	n.s	n.s	n.s	n.s	n.s
MS•LS	n.s	n.s	n.s	n.s	n.s	n.s	n.s	n.s	n.s
LM•LS	n.s	n.s	n.s	n.s	n.s	n.s	n.s	n.s	n.s
RMS•RLM	n.s	s	n.s	n.s	n.s	n.s	n.s	n.s	n.s
RMS•RLS	n.s	s	n.s	n.s	n.s	n.s	n.s	n.s	n.s
RLM•RLS	n.s	n.s	n.s	n.s	n.s	n.s	n.s	n.s	n.s
Combination•reflux	n.s	n.s	n.s	n.s	s	n.s	n.s	s	s

s: significant, $p < 0.050$; n.s: not significant, $p > 0.050$.
•represents T-test between each item.

DO concentrations. In general, stable and high concentration of DO in the outflow met standards [26] and followed the order M > L > MS > S = LM > LS > RLM > RMS > RLS. Rough and porous quartz sand acted as a trickling filter that improved the oxygen enrichment [27]. Furthermore, previous studies [28,29] showed that when a sufficient supply of oxygen is provided to aerobic heterotrophic bacteria for aerobic degradation oxidation, COD, and BOD_5 removal rates increase.

3.3. Turbidity

Some small particles attached to the quartz sand were washed through the column filter after a filtration period [30]; therefore, turbidity measurements were of particular interest in this study. The values of turbidity (18.47 ± 2.42 nephelometric turbidity units) emulated by cellulose or kaolin were similar to discharges from washbasins and showers [7]. All sizes of quartz sand particles performed better singly than in combination, and the individual reflux was superior to that of combinations. Moreover, the outflow exhibited the lowest turbidity values through small-sized quartz sand particles. The highest turbidity values occurred for RMS. In general, the outflow turbidity values in this experiment were stable for different sizes of quartz sand particles and met the standard. They followed the order M > L > S > LS > MS = LM > RLM > RLS > RMS (Fig. 2b). Columns with similar sized quartz sand particles performed almost as well as mixed particle columns, but the first two size intervals performed noticeably better than the last one. The reason for this most likely lies in the improvement of COD removal efficiency. In terms of turbidity removal efficiency, the filter column performed well. Precipitation and adsorption can reduce the turbidity of sewage and the permeability of the filter [6,18]. Rough and porous quartz sand could absorb and intercept suspended solids well, which may reduce the permeability of quartz sand.

3.4. Chemical oxygen demand

Previous studies showed that in aerobic conditions, the performance of a filter using quartz sand is very efficient at lowering COD [8,27]. The freshly prepared synthetic graywater contained a COD level of approximately 275 mg/L. In this experiment, all refluxes based on combinations performed better than the combinations themselves, and all combinations were superior to filters with similar sizes of quartz sand. Moreover, the outflow from RLM sand exhibited lowest COD concentration, with an average value of 54.9 mg/L, and L showed the highest COD concentration (147.1 mg/L). The corresponding lowest and highest removal rates were 38% and 79%, respectively. The outflow COD concentrations in this experiment were generally stable for different sizes of quartz sand particles and followed the order RLM > RLS > RMS > MS > LS > LM > S > M > L (Fig. 2c). In addition, Table 3 further showed that there were significant differences ($p < 0.05$) between the outflow COD concentrations depending on the different particle sizes. However, there were no significant differences between the combinations or related to the refluxes.

3.5. Biochemical oxygen demand

Fig. 2d shows that the overall outflow BOD_5 concentrations were significantly lower than those of the inflow graywater in quartz sand filters of both single and combined sizes. All combinations performed better than the refluxes based on the combinations, and all refluxes were superior to singly sized quartz sand particles. Moreover, the outflow of LM sand exhibited the lowest BOD_5 concentration (7.0 ± 1.1 mg/L), whereas the L sand had the highest BOD_5 concentration (43.4 ± 4.76 mg/L). Correspondingly, L and the LM had the lowest (56%) and highest (93%) removal rates, respectively. The concentration from the LM met the standard for flushing toilets (10 mg/L), urban irrigation (20 mg/L), and building construction (15 mg/L), while all the reflux concentrations met the standard for urban irrigation [26]. Table 3 also shows that there were no significant differences between the outflow BOD_5 concentrations at each stage. By contrast, there were significant differences between them depending on the combination and the reflux. In general, the outflow BOD_5 concentrations in this experiment for differently sized quartz sand particles were stable under the same conditions and followed the order LM > LS >

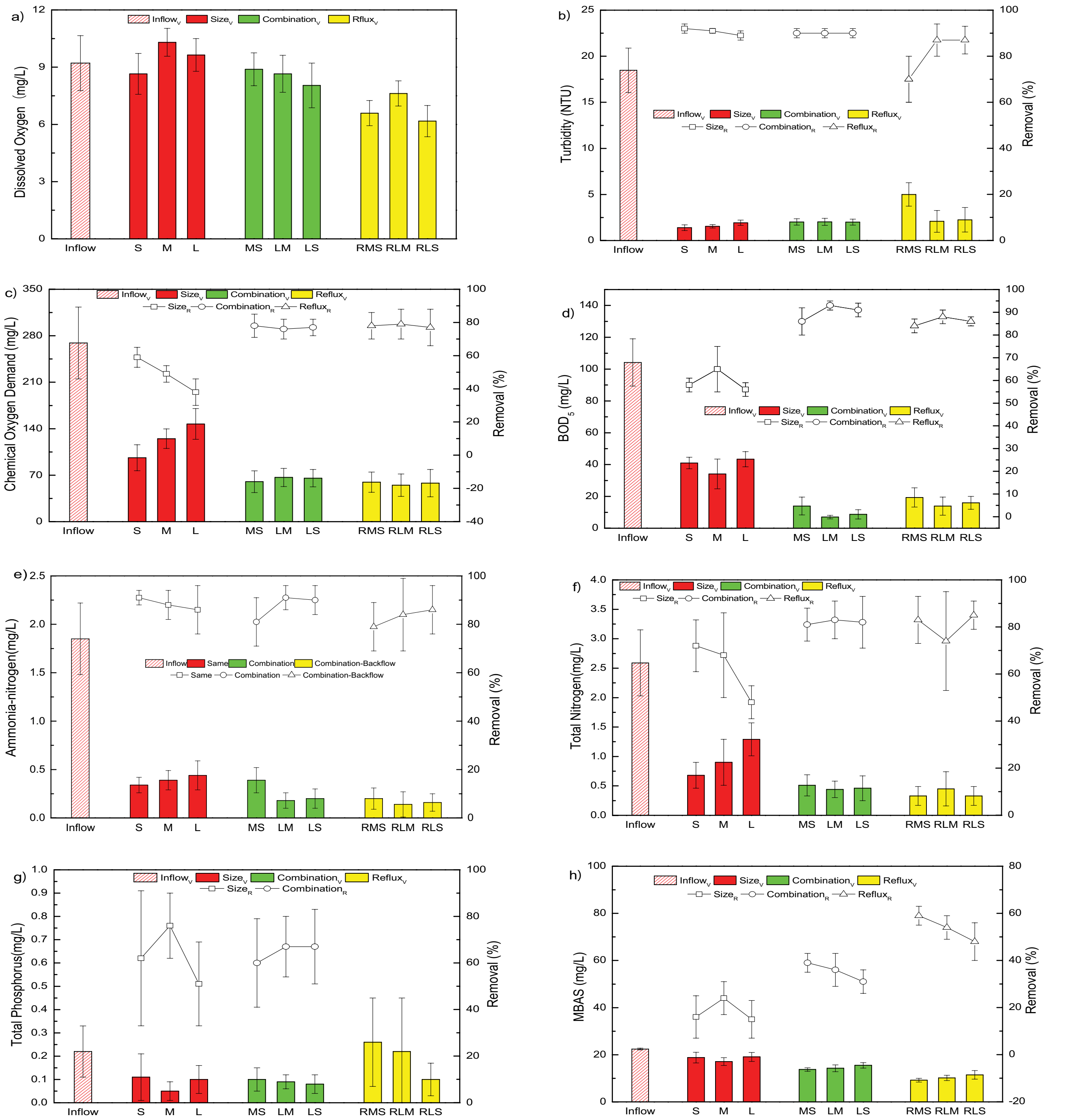

Fig. 2. Comparison between inflow and outflow synthetic graywater treated in a variety of experimental setup designs of quartz sand filters in terms of the following parameters: (a) DO, (b) turbidity, (c) COD, (d) BOD_5, (e) NH_4^+–N, (f) TN, (g) TP, and (h) MBAS. *X*-axis: the number of tested samples was, respectively, 9, 10, and 7 for the three stages. *Y*-axis: the subscripts *V* and *R* refer to the value and removal rate of the water quality index.

MS > RLM > RLS > RMS > M > S > L. Furthermore, the BOD_5 removal rates followed the rank relationship LM > LS > RL M > RLS > RMS = MS > M > S > L (Fig. 2d). Analysis of the inflow graywater also shows that the BOD:COD ratio was approximately 0.5. This could stimulate biological processes and improve treatment efficiencies associated with a lack of nitrogen and phosphorus [31].

3.6. Nitrogen

3.6.1. Ammonia nitrogen

The inflow NH_4^+–N concentration was relatively low according to the recipe used in this experiment (Fig. 2e). The lowest and the highest NH_4^+–N concentrations were found in L and RLM with values of 0.44 and 0.14 mg/L, respectively (Fig. 2e). The corresponding removal ratios were the lowest (79%) in RMS the highest (91%) in LM. In general, the refluxes performed better than the combinations, and all combinations were superior to single sized of quartz sand particles. Overall effluent NH_4^+–N concentrations met the standard for flushing toilets (10 mg/L), urban irrigation (20 mg/L), and building construction (20 mg/L). Moreover, there were no significant differences between the particle sizes, between the combinations, or between the refluxes in terms of NH_4^+–N (Table 3).

3.6.2. Total nitrogen

Inflow TN concentrations were relatively low and their pattern of removal was also very similar to that of NH_4^+–N. The highest and the lowest TN concentrations were obtained in L and RMS, with values of 1.29 mg/L and 0.33 mg/L, respectively (Fig. 2f). The corresponding highest and lowest removal ratios (79% and 91%, respectively) were obtained in RLM and L, respectively. In general, the refluxes performed better than the combinations, all combinations were superior to similar sized quartz sand, and overall effluent TN concentrations met the required standard. The L size and two other sizes showed statistical differences depending on the particle size in terms of TN concentrations. However, there was no significant difference ($p > 0.05$) between the combinations or between the refluxes (Table 3). In addition, the extent of low concentrations of nitrogen and the rough surfaces were suitable for nitrogen recycling, according to previous studies [8,31].

3.7. Total phosphorus

The refluxes did not remove phosphorus efficiently, and the standard deviation of the effluent TP concentrations also fluctuated greatly in the final stage (Fig. 2g). M had the lowest concentration and the highest removal ratio, with values of 0.05 mg/L and 76%, respectively. In general, the combinations performed better than the refluxes, and all combinations showed more stability than similar sizes of quartz sand particles. Owing to the low concentration of the inflow TP, the overall outflow TP met the standard [26]. In terms of NH_4^+–N, TN, and TP, the quartz sand filters had a remarkable effect on lowering the concentrations of nutrients in graywater. Moreover, there were also no significant differences between the TP concentrations in the outflow graywater for each stage, although the refluxes did present a statistically significant difference depending on the size of the sand particle and the combination (Table 3).

3.8. Anionic surfactants

The performance of the ordinary filter using quartz sand was not very efficient at removing anionic surfactants, as shown in previous studies [8,29]. In this experiment, the presence of anionic surfactants in the water quality index was reflected by testing the MBAS of samples. The outflow MBAS of RMS and RLM were 9.21 and 10.17 mg/L, respectively, and their removal ratios were up to 59% and 54%, respectively (Fig. 2h). In general, the reflux based on combinations performed relatively well, and the removal of MBAS followed the rank relationship RMS > RLM > RLS > MS > LM > LS > M > S > L (Fig. 2h). Moreover, there was no significant difference between particle sizes of quartz sand, between the combinations, or between the refluxes in terms of MBAS (Table 3). However, the refluxes based on combinations showed a significant difference with the combinations themselves.

3.9. Microsurface of quartz sand before and after experiments

In previous studies [24,32], SEM was a very effective method to observe the microsurface morphology and structure of the filters and substrates. Fig. 3 shows that there were many pores, sags, and crests on the microsurface of clean quartz sand. Note also that the degree of roughness of each sand particle was evident. When the images were magnified 1,000 times, the degree of roughness followed the order S > M > L. However, when the images were magnified 8,000 times, there were no significant differences in the degree of roughness between three sizes of quartz sand particles. The outflow water quality from the experiments is shown in Table 3 and the outflow for each stage is shown in Fig. 2; these results could confirm to some extent why there were no significant differences ($p > 0.05$) in the outflow water quality from the three filter columns for each stage. Fig. 3 shows that after the second stage, the surface structure of the quartz sand at the same position was similar in the different filter columns. Note also that there were evident changes on the surface of the quartz sand at different positions in the same filter column. The degree of change followed the order MS-u < MS-m < MS-l; LM-u < LM-m < LM-l; and LS-u < LS-m < LS-l. These similar changes further explain why there were no significant differences ($p > 0.05$) in outflow water quality between the different filter columns for each stage. In addition, the clogging of natural porous media often occurs in a wide range of conditions along with physical, chemical, and biological clogging [33]. The results from SEM showed that the substrate in the quartz sand filter became increasingly clogging from the top to the bottom columns. This provides technical guidance and solutions for backwashing.

4. Conclusions

Based on the results of this study, the effects of particle size, substrate combination, and reflux ratio on the quality of the inflow and outflow of graywater were all evident. Substrate combination presented greater differences than the other two combinations. Furthermore, considering the slightly different removal rates, the standard, and backwashing times, the purification performance generally followed the order substrate combination > reflux ratio (1:2) > particle size, and LM (4–8 mm combined with 1–2 mm) was the best substrate combination. It reduced turbidity, BOD_5, and MBAS by up to 90%, 93%, and 36%, respectively. The overall values of outflow pH, DO, turbidity, BOD_5, NH_4^+–N, and TP for LM met the standard for flushing toilets, urban irrigation, and building construction. Thus, this study identified that sand filters can purify graywater efficiently and inexpensively. However, there are other problems with graywater purification using quartz sand filters that need to

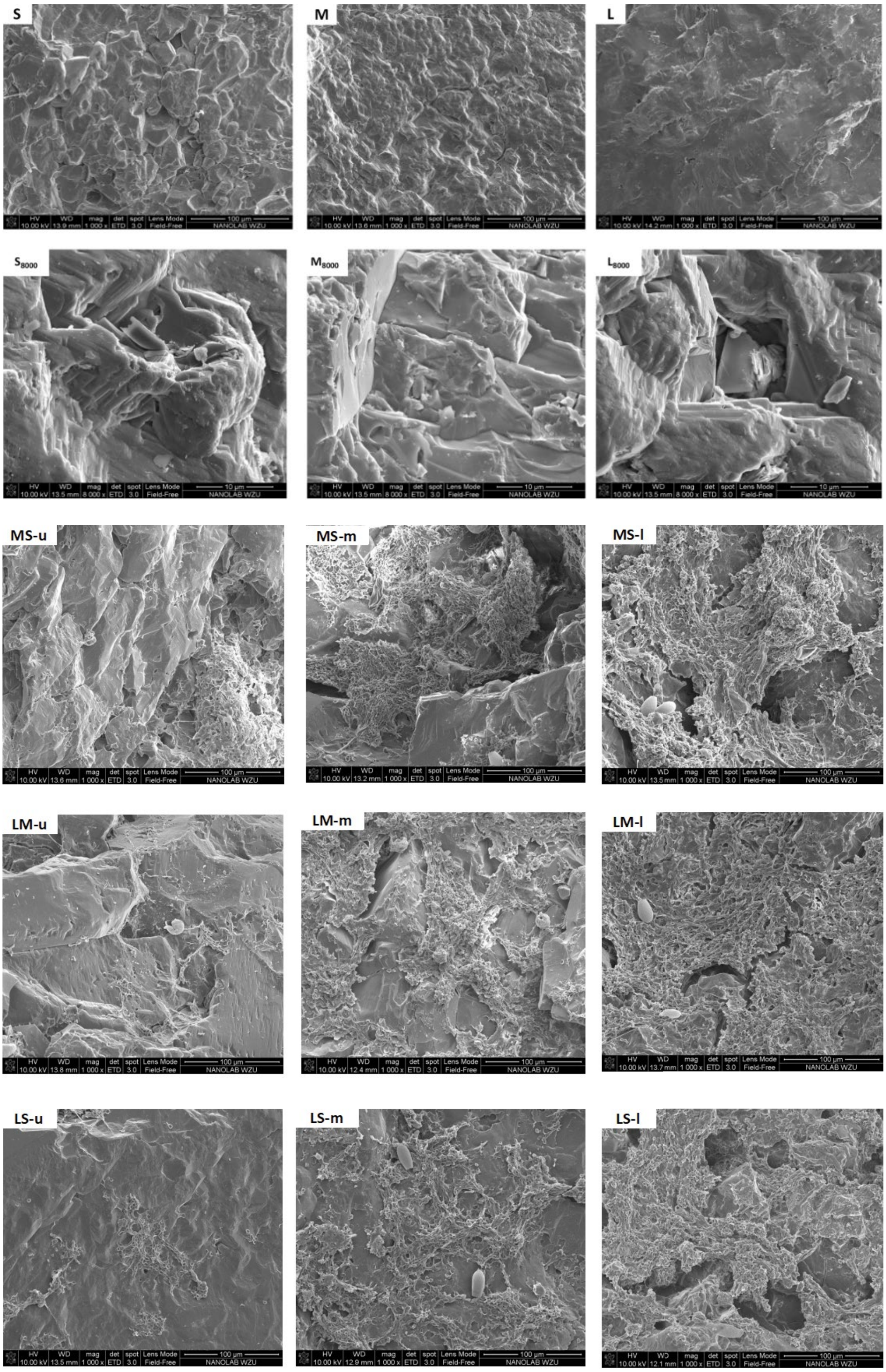

Fig. 3. SEM images of quartz sand. The subscript *u*, *m*, and *l* refer to the upper, middle, and lower quartz sand of the column filter for the second stage, respectively.

be solved, such as understanding the mechanism of removing pollutions and how the removal efficiency of anionic surfactants can be improved.

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