



Removal of zinc from surface runoff by using recycled mussel shell waste as treatment media, with and without heat treatment

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ABSTRACT

Stormwater control measures (SCMs) are essential to manage runoff in urban areas. Mussel shell waste has been recently proposed as sustainable treatment media in SCM to remove metals from runoff. In this study, a group of laboratory-scale column experiments were conducted to investigate the use of crushed mussel shell waste to remove dissolved zinc from actual roof runoff during different filtration flow rates (1, 3, 5, 10 L/min). Heat-treated mussel shells (TMS) and untreated mussel shells (UTMS) were utilized as treatment media with two column depths (1.0 m and 0.8 m). The microstructures and chemical characteristics of TMS and UTMS were examined by using a group of Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS) tests before and after the filtration process, and water samples were analyzed by using an Inductively Coupled Plasma Mass Spectrometry (ICP-MS) instrument. TMS and UTMS showed consistent high removal efficiency for dissolved zinc with (>98%) efficiency during 1 L/min filtration rate. The average removal performance was estimated at >94% and >82% for the 1.0 m and 0.8 m column depths of TMS media, and >92% and >72% for the 1.0 m and 0.8 m depths of UTMS media, respectively. The heat treatment improved the removal of zinc with significant statistical difference (i.e. $p < 0.05$) during short contact times (0.8 m depth, and high filtration rates). Mussel shell waste showed practical removal performance of zinc even during high filtration rates (>5 L/min). Mussel shell waste showed potential benefits as a sustainable and cost-effective filtration media for removal of dissolved zinc in future stormwater systems.

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1. Introduction

Surface runoff is a major source of loading contaminants into urban waterways (Smyth et al., 2021). Contaminants in surface runoff include heavy metals, nutrients, and emerging contaminants such as micropollutants (Müller et al., 2020). Amongst these contaminants, heavy metals, such as zinc, copper, and lead, have detrimental environmental impacts on

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receiving waterways due their toxic effects on freshwater organisms (Davis et al., 2001). For instance, zinc has been classified amongst the contaminants of most concern of urban waterways in Christchurch, New Zealand (Margetts and Marshall, 2018). In particular, dissolved forms of zinc in surface runoff is more difficult to remove by conventional stormwater treatment measures (Charters et al., 2017) and more likely to impacts on aquatic taxa in the receiving waterways.

Contaminant concentrations in runoff are associated with urban surfaces that generate runoff during rainfall events. For example, roof runoff from galvanized material is generally loaded with substantial concentrations of dissolved zinc (Charters et al., 2016) while high concentrations of PAH's and microplastics are usually associated with runoff from roads and industrial sites (Al Ali et al., 2017; Magnusson et al., 2016). Stormwater control measures therefore are essential to manage surface runoff near pollution sources in urban areas.

Different recycled materials such as mussel shell waste have been proposed as sustainable and cost-effective treatment media in stormwater control measures (Bremner et al., 2020). The use of mussel shell waste to remove heavy metals from surface runoff has been investigated in several previous experimental studies (e.g. Bremner et al., 2020; Charters et al., 2021; Good et al., 2014) and different treatment procedures have been proposed to improve the removal performance of mussel shells such as crushing mussel shells into different particle sizes (Craggs et al., 2010) and applying prior heat treatment (Currie et al., 2007).

Previous studies showed high removal efficiency for dissolved metals such as zinc, copper, and lead from surface runoff by using mussel shells (Bremner et al., 2020; Good et al., 2014; Craggs et al., 2010). A recent study conducted by Charters et al. (2021) suggested that the performance of waste mussel shells in removing dissolved zinc from runoff can be as effective as natural materials such as limestone and zeolite. Charters et al. (2021) showed removal of more than 93% of dissolved zinc from roof runoff by using fine-crushed mussel shells (1.18–2.36 mm) in a downpipe treatment system. Another experimental study conducted by Bremner et al. (2020) showed up to 97% reductions in dissolved zinc from synthetic runoff within 48-h of contact time. Another study conducted by Craggs et al. (2010) showed more than 95% reductions in dissolved zinc from synthetic runoff by using crushed mussel shells (i.e. <2 mm particle size) as treatment media in flow-through column experiments within 24-h of retention time.

However, all these studies (summarized in Table A, Supplementary Information) have only investigated the performance of fine-crushed mussel shells (<2.36 mm particle sizes) which only support very low filtration rates (due to low hydraulic conductivity) or extended retention times (batch experiments). Smaller particle size in treatment media provides greater surface area, however it can only tolerate low filtration rates which poses greater risks of clogging and sudden decline in filtration rates. Furthermore, the growth of attached organic matters in mussel shell waste also increases risks of self-clogging in filters, in particular during long retention times and small particle size. For example, Bremner et al. (2020) suggested that attached organic matters on shells reduced its ability to remove dissolved metals, while Charters et al. (2021) reported frequent clogging in treatment media and diminishing hydraulic conductivity overtime.

This study therefore introduces the principle of applying heat treatment to mussel shell waste for two main reasons. Firstly, to remove attached organic matters on mussel shell waste in order to increase the shell's exposure with runoff. Secondly, to prevent/minimize future growth of associated organic matters inside stormwater filters. In fact, it has been suggested that heat treatment can improve the removal performance of mussel shell waste. For example, an experimental study conducted by Currie et al. (2007) revealed that applying heat treatment for fine-crushed mussel shells (i.e. 3 mm–6 mm) resulted in 90% higher removal efficiency of phosphorus from water, comparing to 40% for the untreated mussel shells. This study also aimed to investigate the removal performance with larger particle sizes of mussel shell waste (higher hydraulic conductivity) to allow testing of higher practical filtration rates that have not been examined in previous studies.

Therefore, the first objective of this study was to compare between the removal of zinc from runoff by using heat-treated crushed mussel shells (TMS) vs untreated crushed mussel shells (UTMS). The second objective was to investigate the change in removal efficiency during higher filtration rates and different media depths to represent removal performance during actual conditions.

2. Material and methods

A series of laboratory-scale column experiments were performed to assess the removal efficiency of dissolved zinc by using the TMS and UTMS during different filtration flow rates and contact times.

2.1. Experimental setup

Roof runoff was collected during several rainfall events from galvanized roofing at the Soil and Water Engineering Laboratory at Lincoln University, New Zealand. Actual roof runoff was utilized to represent actual chemistry of runoff quality and to simulate actual treatment performance.

A downpipe was diverted into a 1.0 m³ tank (i.e. Intermediate Bulk Container "IBC") and a peristaltic pump was used to pump the collected runoff into an elevated transparent 1.0 m³ tank in order to run the experiments. The filtration units were made of standard Polyvinyl Chloride (PVC) vessels with an internal diameter of 100 mm connected into a transparent 1.0 m³ PVC tank that contained collected roof runoff (Fig. 1). Two control valves were used to assess the removal performance of zinc during saturated flow rates of 1, 3, 5, 10 L/min. A transparent PVC pipe of 25 mm diameter was utilized as a breathing pipe between the upper valve and the filtration unit in order to release the trapped air in the treatment media and to ensure fully saturated flow conditions throughout the experiments.

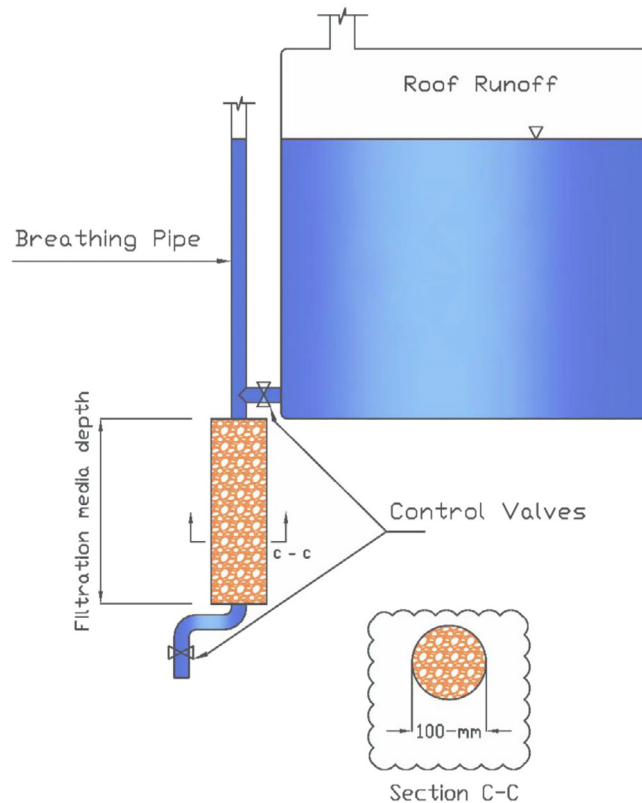


Fig. 1. Experiment configurations used to test the removal performance of TMS and UTMS media.

2.2. Heat treatment and preparation of mussel shells

Partially crushed mussel shell waste was purchased from a local retailer of landscape supplies. Mussel shells were crushed and sieved to the selected particle size (>1 mm and <12.7 mm) to prepare the UTMS (Fig. 2). An additional step of heat treatment was performed to prepare the TMS.

Existing industrial applications apply heat treatment to recycle mussel shells primarily to remove the attached organic and protein matters in the shells (Ballester et al., 2007). Thus, different heat treatment approaches have been introduced to treat mussel shells for different industrial applications. A study by Djobo et al. (2016) revealed that heating the shells at a temperature of $500\text{ }^{\circ}\text{C}$ for 2-h is sufficient to remove all the organic contents. However, a standard treatment method has been suggested by Barros et al. (2009) to treat mussel shells without impacting their chemical compositions which include drying the shells at drying the shell at $190\text{ }^{\circ}\text{C}$ for 18 min and heating for 15 min at $500\text{ }^{\circ}\text{C}$ in order to remove the organic material. In this study, dry crushed mussel shells were available, therefore, the shells were only heated at $500\text{ }^{\circ}\text{C}$ for 15 min (to avoid impacting any chemical composition) and left to cool at room temperature and then re-sieved and washed to remove ashes and fine particles resulted from the heat treatment. The filtration units were then filled with each type of treatment media with similar densities and degree of compactions.

2.3. Characterization of mussel shells

Standard sieve analysis tests were performed to assess particle size distributions of the crushed mussel shells. Chemical characteristics and the microstructures of the TMS and UTMS were assessed before and after the filtration process by using a group of Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS) tests, model no. JEOL JSM-IT300 at the University of Canterbury, New Zealand. The SEM tests were used to take magnified images of the shells and to assess the microstructures of the shells (Fig. 3), and the EDS tests were used to evaluate the chemical compositions for the chemical elements in mussel shell and to evaluate the presence of any suspended zinc particles on the shells after the filtration process (Goldstein et al., 2017).

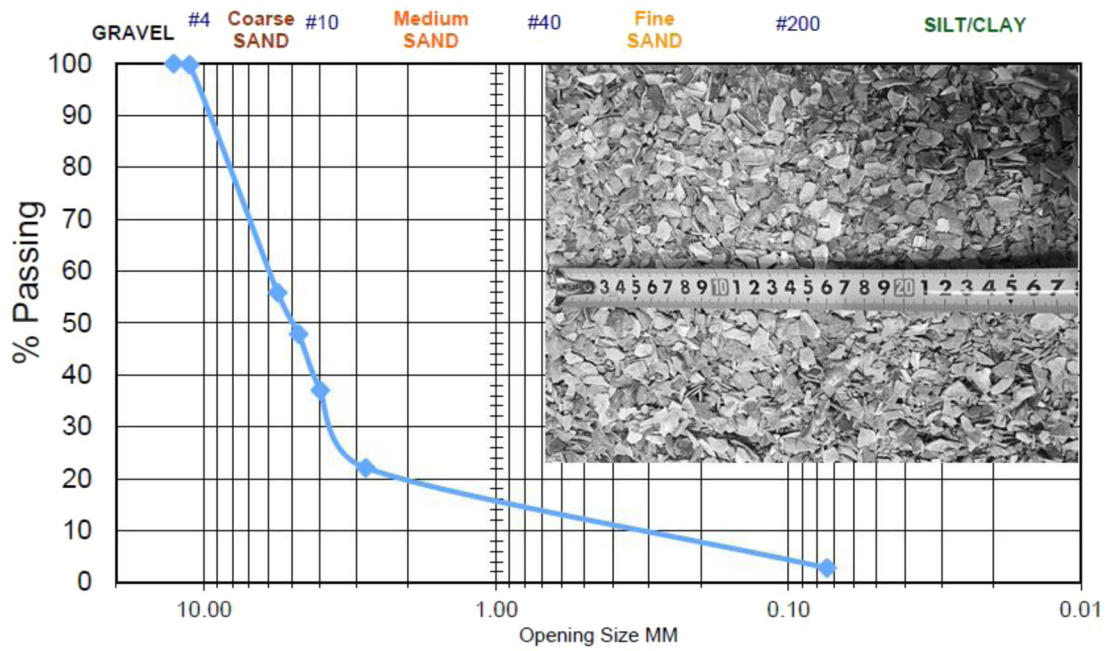


Fig. 2. The particle size distribution for the TMS and UTMS (>1.0 mm and <12.7 mm).

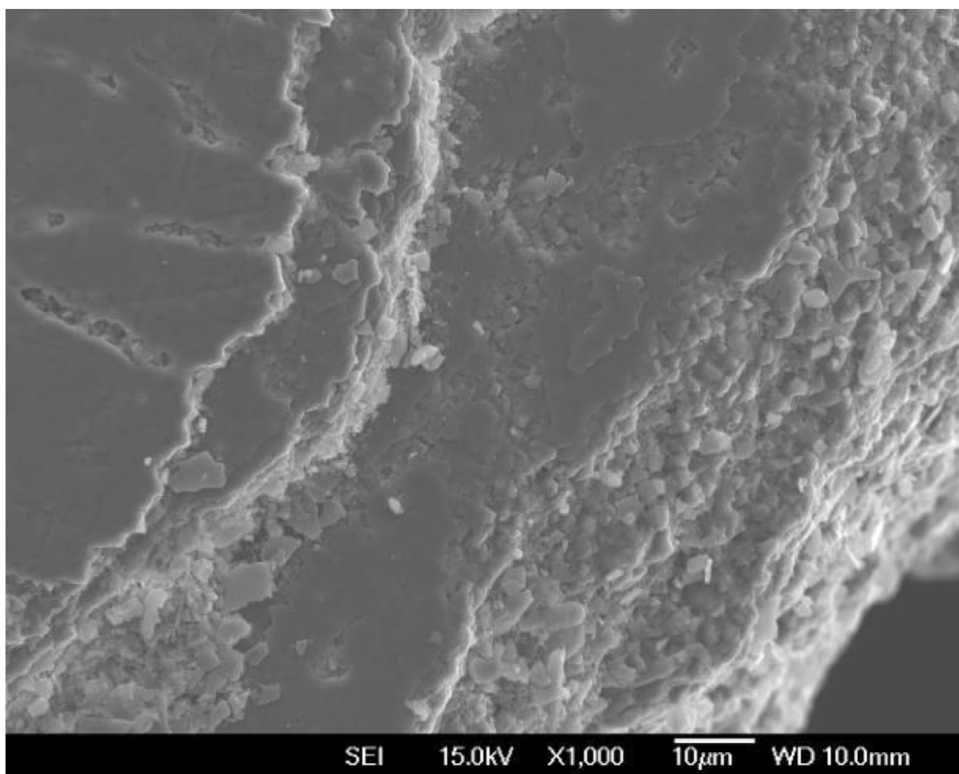


Fig. 3. A SEM image showing the inner and middle layers of the shell. The image shows the tiny particles that form the shell which provide the large contact surface areas with water.

2.4. Running the experiments

The upper and lower valves were fully opened for 1-min to flush the treatment media. The lower valve was then fully closed to fill the filtration unit with water while the upper valve was fully opened. This step continued until the water level in the breathing pipe was stabilized to the same water level as the tank. This step was performed to release all the trapped air via the breathing pipe and to ensure full saturation in the treatment media before running the tests. The lower valve then was partially opened and adjusted to a 1 L/min flow rate and left running for 10 min before taking any water samples. This step was performed to refresh all the water that had longer contact time with the treatment media during the saturation process. The flow rate was then increased to 3 L/min and left running for 3-min. Similarly, flow rate was then increased to 5 L/min and left running for 2-min, and finally to 10 L/min and left running for 2-min. During each flow rate, plastic beakers of 500 ml volume were used to collect the samples from treated water. The samples were collected, and the pH was measured at the end of each flow rate setting. The whole process was repeated three times ($n = 3$) for each type of treatment media.

All samples were sampled by following method 3030-B (APHA, 2005). The collected samples were directly filtered by using 0.45 μm nylon filters into Inductively Coupled Plasma Mass Spectrometry (ICP-MS) tubes and preserved by adding two drops of concentrated nitric acid and stored below 4 $^{\circ}\text{C}$ until delivered to the ICP-MS laboratory for analysis. During testing, the treated water was drained into a 20 L container, and a pH probe, model no. HQ40D Portable Meter, was used to measure the pH readings during testing. Random duplicates and blanks were included in every batch of samples to the ICP-MS to detect any sources of error (e.g. human or instrumental errors) and to improve the precision of results analysis. The t-Test Paired Two Sample for Means method was used to perform the statistical analysis of results (i.e. $p = 0.05$).

3. Results and discussion

3.1. Characteristics of mussel shell

SEM/EDS tests showed similar microstructure and chemical elements in both TMS and UTMS media before and after the treatment process (Table 3). The observed microstructure during SEM tests aligned with previous literature regarding the three layers that constitute mussel shells (Mo et al., 2018).

The outer layer (periostracum) was a green-papery cover and composed of proteins without any minerals. This outer layer appeared to have the least contribution in the removal process of zinc because most of this layer peeled off during the crushing process in the UTMS, and the rest was burned during the heat treatment process of the TMS. The middle layer (prismatic) is the main thick layer of the shell which consisted of parallel calcite prisms oriented 30° toward the outer layer. This part of the shell is perhaps the main contributor to the removal process of dissolved metals due to its special shape and orientation. The multi-arrays prisms of CaCO_3 in the middle layer provided substantial contact surface area with water (Fig. 3). The inner layer (i.e. nacre) is generally classified as a biomineralized layer with 5% of proteins (Gao et al., 2015). Thus, the heat treatment for TMS generally removed proteins and organic matters from this layer which provided larger contact surface areas of CaCO_3 to water.

3.2. Zinc in collected roof runoff

The collected roof runoff showed substantial concentrations of dissolved zinc that ranged between 3071–3801 $\mu\text{g/L}$ with an average concentration of 3347 $\mu\text{g/L}$. This concentration is 200 times higher than recommended concentrations of zinc (15 $\mu\text{g/L}$) in urban waterways according to the Australian and New Zealand Environment and Conservation Council standards (ANZECC, 2000).

It has been long known that influent concentration largely influence treatment performance for dissolved metals. For example, sodium and magnesium ions found in runoff can compete with free metal ions for sorption sites in treatment media (Ziyath et al., 2011). However, the use of actual roof runoff in this study aimed to represent actual chemistry of runoff and consequently reflected actual removal performance during field conditions.

3.3. pH changes

The pH levels in roof runoff ranged between 6.35–6.97. Both TMS and UTMS increased pH levels in runoff. TMS media increased pH levels from 6.75 to 8.77, while UTMS media showed average increase from 6.75 to 8.24 during all tested filtration rates.

The increase in pH for treated runoff supports previous findings that suggest higher removal performance of zinc at high pH levels (Barakat, 2008). This is due to the natural composition of CaCO_3 in mussel shells which causes the pH of runoff to increase, and consequently reduces the solubility of zinc (Hu et al., 2011).

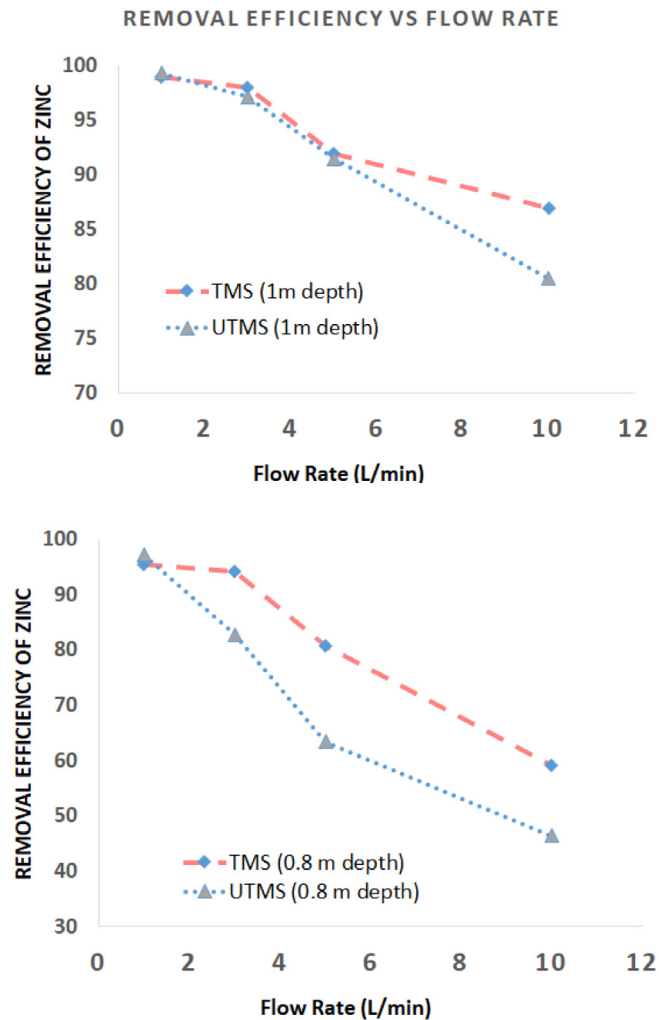


Fig. 4. The removal efficiency vs flow rate of the TMS and UTMS in 1.0 m, 0.8 m of treatment media.

Table 1

The dissolved zinc concentrations ($\mu\text{g/L}$; mean \pm SD), and the Removal performance (%; mean \pm SD) for the TMS and UTMS with 1.0 m depths of treatment media. Flow rates = 1, 3, 5, 10 (n = 3).

Flow (L/min)	TMS		UTMS	
	Zinc ($\mu\text{g/L}$)	Removal performance (%)	Zinc ($\mu\text{g/L}$)	Removal performance (%)
Untreated runoff	3582.95 \pm 308	-	3582.95 \pm 308	-
1	45.65 \pm 8.88	98.80 \pm 0.23	20.72 \pm 3.05	99.38 \pm 0.1
3	43.24 \pm 4.53	98.86 \pm 0.12	94.39 \pm 8.61	97.19 \pm 0.26
5	341.13 \pm 40.92	91.03 \pm 3.71	285.18 \pm 71.34	91.52 \pm 2.12
10	421.29 \pm 46.86	88.92 \pm 1.23	656.18 \pm 132.05	80.45 \pm 3.92

3.4. Removal efficiency of zinc

Both TMS and UTMS media showed high removal performance for dissolved zinc. The average removal of zinc during all tested flow rates was estimated at $>94\%$ and $>82\%$ for 1.0 m and 0.8 m column depths of TMS media, and $>92\%$ and $>72\%$ for 1.0 m and 0.8 m depths of UTMS media, respectively (Tables 1 and 2). Removal performance of zinc decreased as water flow through the TMS and UTMS increased (Figs. 4 and 5).

TMS and UTMS showed direct correlations between the removal performance of dissolved zinc and the contact time with water. The increase in flow rates through the treatment media (shorter contact time) resulted in lower removal performance, while for similar flow rates, the increase in treatment media depths (i.e. longer contact time) showed higher removal performance. However, the removal performance of zinc was consistent during each flow category. Such removal

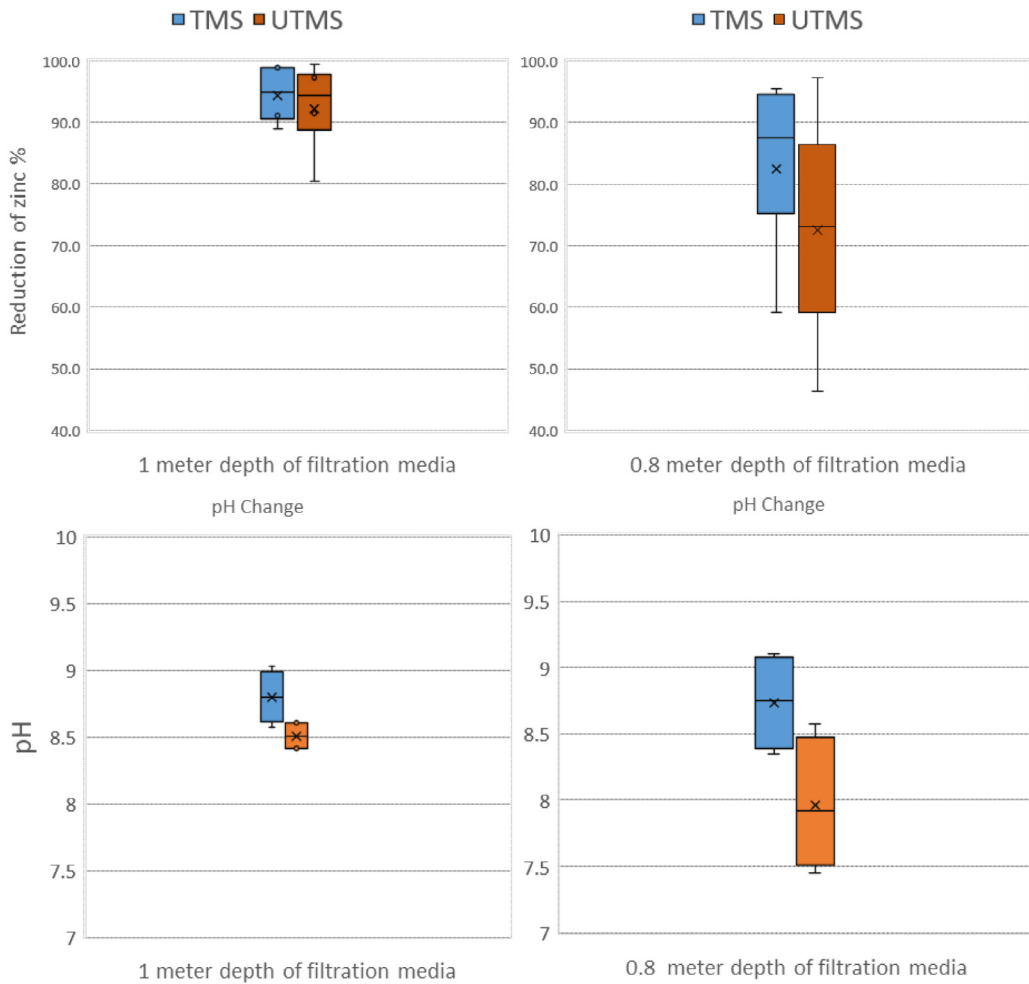


Fig. 5. The reduction of zinc concentration (%) and pH change in runoff for the 1.0 m, and 0.8 m column-depths of the TMS and UTMS.

Table 2

The dissolved zinc concentrations ($\mu\text{g/L}$; mean \pm SD), and the removal performance (%; mean \pm SD) for the TMS and UTMS with 0.8 m depths of treatment media. Flow rates = 1, 3, 5, 10 (n = 3).

Flow (L/min)	TMS		UTMS	
	Zinc ($\mu\text{g/L}$)	Removal performance (%)	Zinc ($\mu\text{g/L}$)	Removal performance (%)
Untreated runoff	3111.66 \pm 59	-	3111.66 \pm 59	-
1	141.6 \pm 28.9	95.4 \pm 0.94	87.9 \pm 13.5	97.2 \pm 0.42
3	178.4 \pm 28.1	94.2 \pm 0.91	541.9 \pm 60.64	82.8 \pm 1.92
5	593.9 \pm 114.19	80.7 \pm 3.72	1155 \pm 185.94	63.4 \pm 5.9
10	1252.3 \pm 150.17	59.2 \pm 4.89	1690 \pm 125.31	46.4 \pm 3.97

performance aligns with previous findings in other experimental studies (e.g. Bremner et al., 2020; Charters et al., 2021; Good et al., 2014).

During low filtration rates (1 L/min) and 1.0 m media depths (longer contact time) TMS and UTMS showed similar removal performance of zinc with averages of 97%, and 98% respectively. However, during high filtration rates (10 L/min) and 0.8 media depths (shorter contact times) TMS media showed higher removal efficiency with an average of 74% comparing to 63% for UTMS. Thus, TMS media showed higher removal performance with significant statistical difference (i.e. $p < 0.05$) during higher filtration rates and 0.8 m depth of treatment media.

Previous studies identified either adsorption, precipitation (filtration), or a combination of both as the main removal mechanisms of dissolved metals by using mussel shells as treatment media (Good et al., 2014; Bremner et al., 2020). In this study, observed high removal efficiencies at pH levels of treated runoff support the theory that high pH levels reduce the solubility of zinc (Hu et al., 2011). However, low presence of zinc particles on the tested shells support the theory that

Table 3
Individual chemical characteristics
(mean \pm SD Wt %) of mussel shells
(n = 4).

Mussel shell	
Element	Wt %
Oxygen	51.41 \pm 9.5
Calcium	44.96 \pm 12.82
Silicon	0.93 \pm 0.76
Sodium	0.96 \pm 1.2
Aluminum	0.84 \pm 0.84
Sulphur	0.67 \pm 1.05

adsorption is perhaps the main removal mechanism of dissolved metals and suggest that precipitation or a combination of both as secondary removal mechanisms.

Understanding of the mechanisms and kinetics of mass transfer by using the adsorption kinetic models may help advance the technology of applying waste mussel shell to pollutants in future stormwater technologies. Thus, future in-depth research should focus on creating adsorption kinetic models (e.g. [Fulazzaky et al. \(2021\)](#)) along with geochemical modeling to identify key removal mechanisms and interactions between water and mussel shells.

3.5. Heat treatment and removal performance

The applied heat treatment on mussel shell waste improved its ability to remove zinc from runoff. The higher removal performance achieved by using TMS (without organic content) supports previous literature that suggested adsorption to CaCO_3 surfaces as the primary metal removal mechanism in the mussel shells ([Barakat, 2008](#)).

The presented results in this study indicate that heat treatment for mussel shells is perhaps a way to improve the shell's performance to remove heavy metals from runoff. Removing organic matters from mussel shells allow larger surface areas of CaCO_3 to be exposed to water. In the same context, [Bremner et al. \(2020\)](#) suggested that the presence of biofilms in treatment media (organic material) might reduce the removal efficiency of dissolved heavy metals from water.

Heat treatment is primarily performed to remove attached organic matters ([Ballester et al., 2007](#)) and to prevent the growth of associated biofilms in stormwater filters. In this study, the heat treatment reduced the content of organic matter in mussel shells (strong smell of burned organic matters was observed during the heat treatment). However, removing all organic matter might require higher temperatures and longer heating times ([Djobo et al. \(2016\)](#)).

4. Conclusion

This study compared between the use of heat-treated and untreated mussel shells as treatment media in stormwater control measures. Both TMS and UTMS showed high removal efficiency for dissolved zinc during low filtration rates, and heat treatment further improve the removal efficiencies during high filtration rates (short contact times). Heat treatment removed organic matters attached to mussel shells and consequently exposed larger surface areas of CaCO_3 to water. This helped to increase the removal performance and minimized clogging issues caused by the growth of associated organic matters inside stormwater filters. Finally, mussel shell waste showed a potential opportunity to provide a sustainable and cost-effective treatment media by recycling giant piles of unwanted waste from shellfish industries into stormwater treatment industries.

Future studies to optimize mussel shell performance in treatment technology should address the following research questions:

- What are the main removal mechanisms and mass interactions between water and mussel shell waste?
- How different heat treatment procedures (higher temperatures or longer heating times) impact the removal performance of mussel shells?
- What is the long-term removal performance of mussel shells under continuous flow conditions? Expected lifespan of treatment media before breakthrough?
- What is the relationship between mussel shell particle sizes and removal performance during similar flow conditions?
- What is the optimal particle size of mussel shell waste that provides maximum removal performance?

CRedit authorship contribution statement

Mohamad Odeh: Conceptualization, Methodology, Investigation, Data curation, Writing – original draft. **Crile Doscher:** Supervision, Conceptualization, Methodology, Writing – review & editing. **Thomas A. Cochrane:** Supervision, Conceptualization, Methodology, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary material related to this article can be found online at <https://doi.org/10.1016/j.eti.2022.102814>.

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