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Facile Fabrication of Water Filters with Desirable Properties for Removal of Pathogenic *E. coli* from Drinking Water at point-of-use

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Abstract

The inevitable consequences of waterborne diseases caused by consuming contaminated water have prompted the development of low-cost water filtration technologies that surpass traditional purification methods in effectiveness. Ceramic water filters have been extensively researched due to their promising application in the filtration and purification of water to produce clean and safe drinking water. The present study reports the synthesis of ceramic filtration systems incorporating silver nanoparticles with desirable properties for water purification. The obtained samples were examined by XRD, XRF, and SEM analyses. XRD analysis showed that the samples had crystallite sizes ranging between 16 to 22 nm. XRF analysis revealed a high percentage of SiO_2 (94%) contributed by the high silica content from sand and rice husk. SEM analyses showed the formation of mainly spherical aggregates. The efficiency of the obtained samples was tested by their performance in filtering pond and river water. Prior to water treatment, *E. coli* counts and other physiochemical parameters were measured. The colorimetry results on *E. coli* removal by the prepared samples demonstrated good performance of up to 99.999% for samples of ratio 60:40:7.5 which were sintered at 850 °C. These results indicate that Ag-NPs/CWF samples are capable of removing a variety of pollutants, particularly *E. coli* and other physiochemical parameters making them a promising solution for water disinfection and purification.

Key Words: Ceramic filters; Silver nanoparticles; Waterborne diseases; Water disinfection; Calcination

Introduction

Accessibility to reliable and safe drinking water is the challenge facing the world, especially developing countries. Over 40% of the population does not have access to sufficiently clean and safe drinking water (WHO 2019). A 2023 report from the World Health Organization (WHO) and United Nations Children's Emergency Fund (UNICEF's) Joint Monitoring Programme for Water Supply and Sanitation highlights that 2 billion individuals do not have access to safely managed drinking water services, while 3.6 billion people are without safely managed sanitation services (Dickin & Gabrielsson 2023, Girmay et al. 2023). Bacterial contamination, particularly *E. coli* contamination of drinking water sources is the greatest hazard to human health, leading to outbreaks of numerous waterborne diseases (Khan et al. 2021, Kristanti et al. 2022). Most of the waterborne diseases in many parts of the world are caused by the use of contaminated water and poor sanitation (Shayo et al. 2023). This case is even more pronounced in developing countries (Kilungo et al. 2018, Mukaratirwa-Muchanyereyi et al. 2020). Specifically, the UN report released in 2019 disclosed that about 2.1 billion people throughout the world still lack access to safe drinking water. As a result, 2 million people die each year from waterborne diseases, and children under the age of five are the major victims (UN 2019).

Access to clean drinking water poses a significant challenge for the majority of individuals in developing nations due to the frequent absence of municipal water treatment systems. Nevertheless, Tanzania has demonstrated noteworthy advancements in enhancing basic drinking water services for its populace in recent years, as indicated by Kilungo et al. (2018) and TDHS (2019). In 2010, merely 32% of Tanzanians had access to safe drinking water. This figure rose to 50% in 2015 and further increased to 61% by 2023, according to WHO (2022), and the World Bank (2023). It is crucial to highlight that while urban areas in Tanzania generally enjoy well-managed access to improved drinking water, the situation in rural areas is notably less regulated, and in some cases, non-existent (Gandidzanwa and Togo 2022, Kilungo et al. 2018). In urban regions, about 88% of enhanced drinking water sources are shielded from fecal contamination, industrial effluents, and other human activities. Conversely, in rural settings, nearly 49% rely on less-managed water sources such as surface water, natural springs, and dug wells (WHO 2022). Unfortunately, these sources are susceptible to contamination from chemicals, pathogens, and physical impurities. The pollution of surface and groundwater is often linked to industrialization, agriculture, mining, and various other human activities (Adesakin 2020, Sarker et al. 2021).

Meanwhile, pathogenic bacteria such as *E. coli* are a biological threat in water (Olatunji et al. 2024, Shayo et al. 2023). The substances such as nitrates, lead, arsenic, pesticides, and radioactive chemicals are chemical hazards that affect the quality of water as well (Ugwu *et al*. 2022). The quality of intended water for home use is of the utmost importance since it must be devoid of elements harmful to human health. Domestic water supplies must be devoid of disease-carrying microbes in order to be safe for human consumption. Pathogens including those that cause intestinal diseases (dysentery, cholera, and typhoid fever) can be transferred by water as well as infectious hepatitis and polio. Water sources in industrialized European and North American nations are largely free of these organisms (Shayo et al. 2023). However, this is not apparent in developing countries, which is why outbreaks of waterborne diseases like typhoid fever and cholera are still not uncommon (Semenza et al. 2022). Nonetheless, even in developed nations, waterborne infections remain a risk, so water sources are frequently tested for fecal coliform bacteria, indicating the presence of fecal discharges in the water.

Ceramic materials have found widespread application in the integration of silver nanoparticles (Ag-NPs) into point-of-use (POU) systems for bacterial removal due to their cost-effectiveness attributable to abundance and local availability. The combination of Ag-NPs with ceramics has displayed considerable efficacy in wastewater treatment (Dong et al. 2022, Oyanedel-Craver and Smith 2008, Assadi et al. 2022 and Yerli-Soylu et al. 2022). Alherek. (2023) observed a substantial decrease in *E. coli* levels through ceramic water filters (CWFs) coated with Ag-NPs, with an average flowing silver concentration of 0.02 mg/L, meeting the drinking-water standards established by WHO and United States Environmental Protection Agency (USEPA) (Dong et al. 2022). Similarly, Oyanedel-Craver and Smith (2008) have reported *E. coli* removal rates ranging from 97.8% to 100%. These studies suggested that factors influencing the release of silver into the filtered water encompass the quantity of silver integrated into the ceramics, the water's chemistry, and the pore structure of the ceramic material.

Due to the consequences brought forth by waterborne diseases from ingesting contaminated water, the synthesis of low-cost water filtration technology that is more effective than the traditional way of purification is inevitable. Various water purification methods have been documented, such as slow sand, ceramic, bio-sand, membrane, and bone char filters (Shayo et al. 2024). Among these methods utilized in different places, ceramic water filtration systems are regarded as a low-cost technology since their synthesis process employs the use of inexpensive and readily available materials from the environment, such as sawdust, wheat flour, starch, rice husk, and clay/soil (Shayo et al. 2023, Singh et al. 2021). Many countries have used ceramic-based water treatment technology which presented to having good results (Dong et al. 2022). The studies are reporting that ceramic filters can be incorporated with nanoparticles to enhance their performance in water purification.

Recently, the use of nanotechnology for water treatment has sparked a lot of attention (Jain et al. 2021, Singh et al. 2022). In particular, noble metal nanoparticles such as silver, gold, zinc, and copper can be employed for small-scale or point-of-use water systems, particularly in removing pathogenic bacteria (Joshi et al. 2021; Zhao et al. 2020). However, in Tanzania, little has been preserved in practice and the available market filter products lack practical validation under local conditions (Shayo et al. 2024, Tariq et al. 2023). This is because available ceramic filters can barely remove other water contaminants and are ineffective at removing *E. coli* contaminants from drinking water at high rates. Therefore, there is a great need to fabricate functional ceramic-based water purification systems that suit Tanzania's settings.

According to Nakamura et al. (2019), direct contact between silver and a contaminated organism's cell wall is the main method used by Ag-NPs. The dissolution of bacterial cell membranes and the release of Ag^+ ions are the first steps in this process. Silver ions interact with respiratory and transport proteins because of their high affinity for them. Godoy-Gallardo et al. (2021) suggested that silver ions can alter deoxyribose nucleic acid (DNA) preventing bacterial growth and replication. Furthermore, metals can function as catalysts to produce Reactive Oxygen Species (ROS) when dissolved oxygen is present. Ag-NPs may improve interactions with oxygen which could increase the production of free radicals on their surface and lead to membrane damage (Godoy-Gallardo et al. 2021). The present study introduces the cost-effective method to fabricate silver nanoparticles incorporated with ceramic water filters (Ag-NPs/CWF) using locally available materials such as sand, wheat flour, and rice husk. Silver nanoparticles were incorporated into the CWFs to enhance their performance in removing bacterial contaminants such as *E. coli*. Moreover, the performance of the obtained ceramic materials was tested in filtering water collected from Itamba Pond and Ruaha River (Kalenga), to establish their practical reliability in purifying contaminated water.

Materials and Methods

This investigation aimed to design, fabricate, and evaluate the efficiency of CWFs. The experiment employed readily available materials: sand, rice husks collected from a local rice milling industry in Iringa, and wheat flour. Analytical grade chemicals: $AgNO_3$ (Sigma-Aldrich), $NaBH_4$ (Merck), and HNO_3 (Fisher Scientific), were purchased and utilized without further purification. Additional resources included Coleman cool boxes for preserving water samples collected from the Ruaha River and Itamba ponds. In a typical experiment, rice husk ashes (RHA) were made by burning rice husks at 600°C in a muffle furnace, heating them slowly at a rate of 5°C per hour. This process was done to get a high amount of silica from the rice husks. The ceramic mixture, made from sand and RHA, was mixed in a 60:40 ratio (sand to RHA). Then, different amounts of ceramic fillers (wheat flour) of 7.5%, 15%, and 30% were added to this mixture to obtain samples dubbed as 60:40:7.5; 60:40:15 and 60:40:30, respectively. These ingredients were then mixed with 100 mL of distilled water and stirred for 2 h. The samples were filtered using a vacuum pump and then dried in an oven at 80 °C followed by calcination at two different temperatures (650 °C and 850 °C).

Silver-incorporated ceramic materials were prepared by dissolving 0.4 g of $AgNO_3$ into 100 mL of distilled water, followed by 4 g of the prepared ceramic sample. The sample was stirred for 30 minutes to form a homogeneous solution (Solution 1). On the other hand, 0.4 g of $NaBH_4$ was dissolved in 150 mL of distilled water and the solution was stirred for 30 minutes (Solution 2). Using a burette, solution 2 was added dropwise to solution 1, and the mixture was stirred for 2 hours. The mixture was filtered using a vacuum pump and dried at 80 °C to obtain the final product. The obtained sample was calcined in a muffle furnace for 3 h at 600 °C.

An X-ray diffractometer (D2-PHASERE A-26-X1-ABOD2C) was used to investigate the crystalline properties of the obtained samples using Cu Kα radiation (λ=1.5406 Å). The accelerating voltage and applied current were 40 kV and 100 mA, respectively. The crystallite size of the powders was estimated using the Scherer equation reported in the literature (Fatimah et al. 2022). Field-emission scanning electron microscopy (FE-SEM, MIRA-3 Tescan) with an accelerating voltage of 15.0 kV was used to study the morphology of the samples. Bulk elemental analysis of the samples was performed by an X-ray fluorescence spectrometer (XRF; Thermofiscers ARL 9900 dispersion wavelength spectrometer which was obtained at SEAMIC with a detection limit of 10 ppm and depth resolution of up to 10 µm). ZAF corrections (atomic number, Z) were used to determine the molar ratios of the elements present in the samples.

The powdered samples (15 g of synthetic Ag-NPs/CWF) were loaded onto white cotton silk cloth material. The sampled water (which is a primal source by the settles of the sampled areas) was then allowed to pass through to test the efficiency of the designed samples for *E. coli* removal from water, which was then analyzed by a colorimeter. Prior to and after the sampled water was passed into the synthesized samples, the parameters of that water were measured in triplet. The results were then compared to measure the effectiveness of the Ag-NPs/CWF in removing *E. coli* from the water and the best-obtained results were reported.

Results and Discussion

The x-ray diffraction (XRD) patterns of the samples obtained at different conditions are presented in Figures 1, 2, and 3.

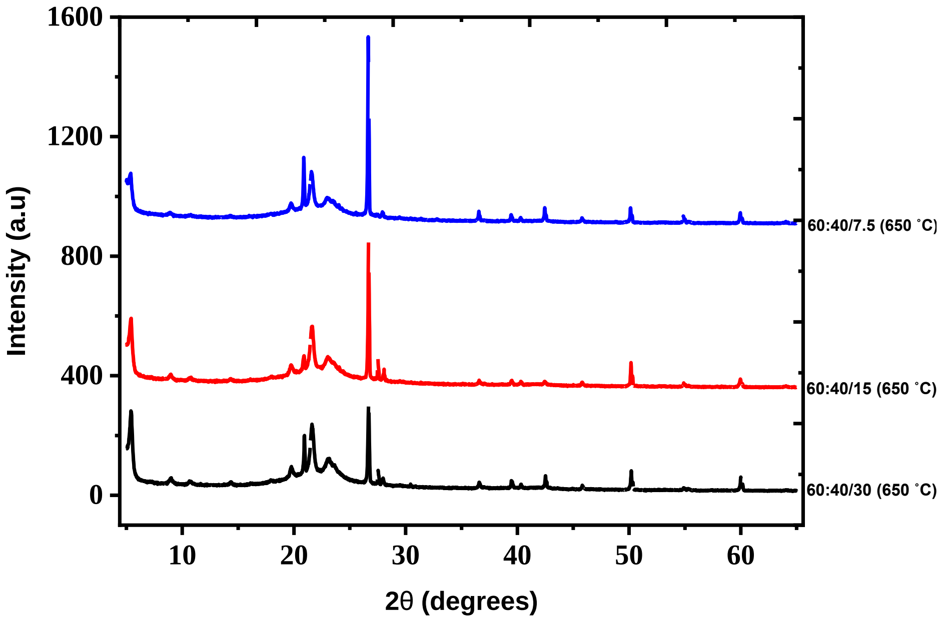

Figure 1: XRD patterns of the prepared ceramics at different ratios and calcined at 650 °C

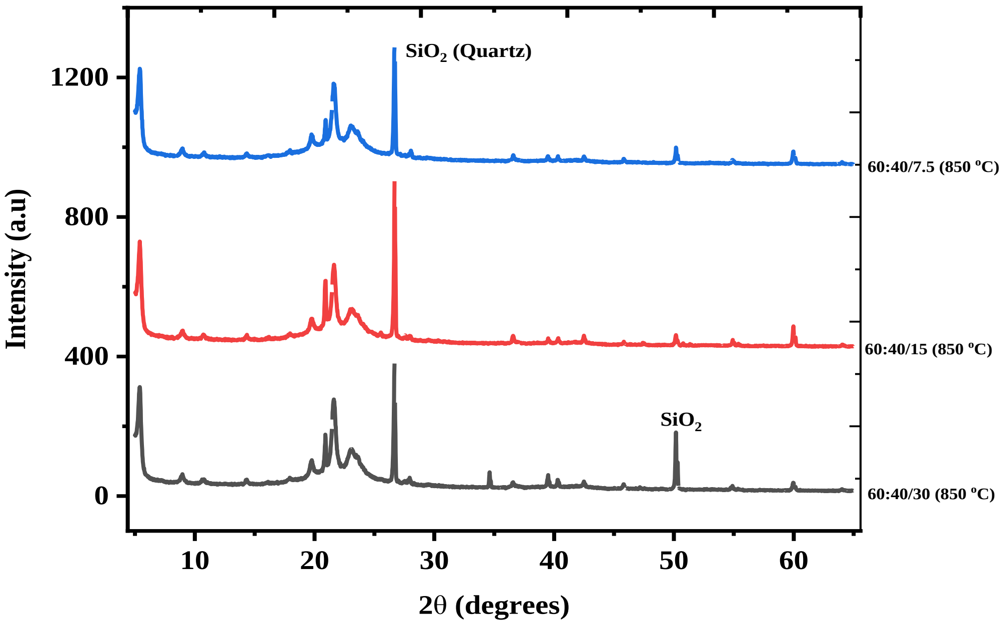

Figure 2: XRD patterns of the prepared ceramic at different ratios and calcined at 850 °C

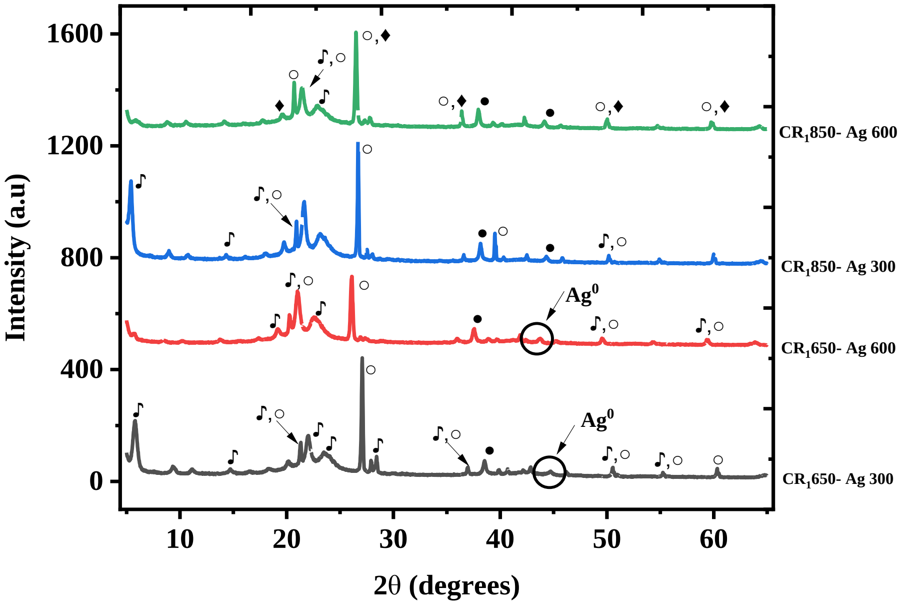

Figure 3: XRD patterns of ceramic products synthesized for ratio 60:40:7.5 and sintered at temperatures of 650 °C and 850 °C and calcined at different temperatures of 300 °C and 600 °C after $AgNO_3$ addition, where SiO_2 peaks ○- quartz, ♪- Tridymite, ♦- Moganite, and ●- Ag^0

In addition, Figure 3 presents the samples analyzed at two different sintering temperatures 650 °C and 850 °C. The figure illustrates the crystal structure of the sample synthesized with a ratio of 60:40:7.5 (referred to as CR1). Figures 1 and 2 show that all SiO_2 peaks associated with various types and phases as presented in Figure 3. However, with the addition of silver to the ceramic samples, new peaks appear at 2θ angles: 38.3°, 44.4°, and 64.4°. These peaks correspond to the lattice plane clusters (111), (200), and (220), respectively (Dung et al. 2019, Ali et al., 2023, Mukaratirwa-Muchanyereyi et al. 2019). The presence of these peaks indicates the presence of silver nanoparticles in the sample. These studies report that Ag^0 can infect and remove *E. coli*, which are waterborne and pathogenic microorganisms.

Several studies have reported the thermal reduction of silver ions (Ag^+) into silver metallic (Ag^0) form (Ali et al. 2023, Njoki et al. 2020). As reported in a similar study, SiO_2 quartz (hexagonal) has strong diffraction peaks at 2θ ° of 21.6, 26.6, and 50.1, followed by aluminum silicate (orthorhombic mullite) with strong diffraction peaks at 2θ ° of 16.4, 30.9, 33.1 and 40.8. In the present study, the results show that the reduction of silver nitrate-impregnated ceramic samples to silver metal (Ag^0) during the firing process was successful. According to Ali et al. (2023) in a similar study, Ag^+ is reduced to Ag^0 at 600 °C, and these Ag-NPs can then be oxidized into Ag^+ at an elevated temperature of 1000 °C. In addition, some of the common peaks were observed in all figures (1, 2, and 3) which include a minor aluminum oxide phase with diffraction peaks at 2θ ° of 16.4, 30.9, 33.1, and 40.8, SiO_2 quartz which is a significant phase with strong diffraction at 2θ ° of 20.8, 26.6 and 50.1. The formation of feldspar or plagioclase phases, which can occur between 650 and 850 °C, could be the cause of the minor peak observed at the 2θ ° of 27.5 (Annan et al. 2018). Therefore, this implies that Ag-NPs can be incorporated into the CWF samples as reported in similar studies done by Mukaratirwa-Muchanyereyi et al. (2019) and Dung et al. (2019).

X-ray fluorescence analysis was used to study the elemental composition of the samples. It can be seen that the samples contained high SiO_2 (82%) and low MnO (0.02 %), while Ag-NPs were also observed as indicated in Table 1 particularly in CR_1 850- 600 °C, CR_2 850- 600 °C and CR_3 850- 600 °C.

Table 1: The elemental analysis by XRF from the synthesized ceramic samples.

Element (s)	**Sand**	**RHA**	**60:40/30 (850 °C)**	**60:40/15 (850 °C)**	**60:40/7.5 (850 °C)**	**CR_1 850-Ag 600 °C**	**CR_2 850-Ag 600 °C**	**CR_3 850-Ag 600 °C**
SiO_2	70.10	84.11	8.76	82.92	94.33	75.54	78.37	79.91
Al_2O_3	6.18	0.06	2.48	1.04	1.27	1.79	1.19	0.99
Fe_2O_3	3.27	0.06	5.50	0.78	0.78	0.72	0.89	0.90
CaO	17.18	13.80	81.82	13.40	0.78	12.34	17.80	15.84
Ag_2O	0.01	0.00	0.00	0.01	0.03	0.81	0.95	0.78
MgO	0.25	0.26	0.37	0.27	1.40	0.18	0.16	0.18
K_2O	1.57	0.46	0.17	0.77	0.28	2.59	0.20	0.67
Na_2O	0.66	0.77	0.04	0.17	0.58	0.27	0.02	0.29
SO_3	0.08	0.25	0.17	0.21	0.17	0.36	0.18	0.16
MnO	0.02	0.02	0.04	0.02	0.02	0.54	0.02	0.04
P_2O_5	0.02	0.12	0.00	0.23	0.18	3.94	0.02	0.02
TiO_2	0.66	0.08	0.66	0.18	0.18	0.72	0.22	0.22
TOTAL	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**

The silica (SiO_2) content was notably higher due to the materials used in the study, specifically sand, which contains over 90% silica, and rice husk ash, which also comprises more than 90% silica. Regarding heavy metal elemental compositions, the synthesized samples contained Fe_2O_3, MnO, TiO_2, and Ag_2O, though their amounts were quite small, at ≤ 0.9 mass percent, except for Fe_2O_3, which had a higher percentage of about 5.5% from the sand sample. Other detected elements included CaO, which followed SiO_2 in abundance, as well as Al_2O_3, MgO, K_2O, Na_2O, SO_3, and P_2O_3.

The SEM images of the prepared samples are displayed in Figures 4 (a) - (f). Generally, these samples display different morphologies depending on the presence or absence of Ag nanoparticles. The samples were synthesized in ratios of 60:40:7.5 (a-b), 60:40:15 (c-d), and 60:40:30 (e-f). Figure 4 (a), (c), and (e) samples without AgNPs, while (b), (d), and (f) are ceramics with AgNPs. The SEM images in Figure 4 are comprised of mainly irregular and compact spherical aggregates. The porous system in Ag-NPs/CWF samples consists of numerous pores and channels with a diameter or width of up to several micrometers as can be seen in the SEM images. Water flows through these macropores in the ceramic filter and expels large suspended particles. For better performance and filter lifespan, suspended particles were removed from the water before filtration. However, in Ag-NPS/CWF, mesopores also play a significant role during filtration.

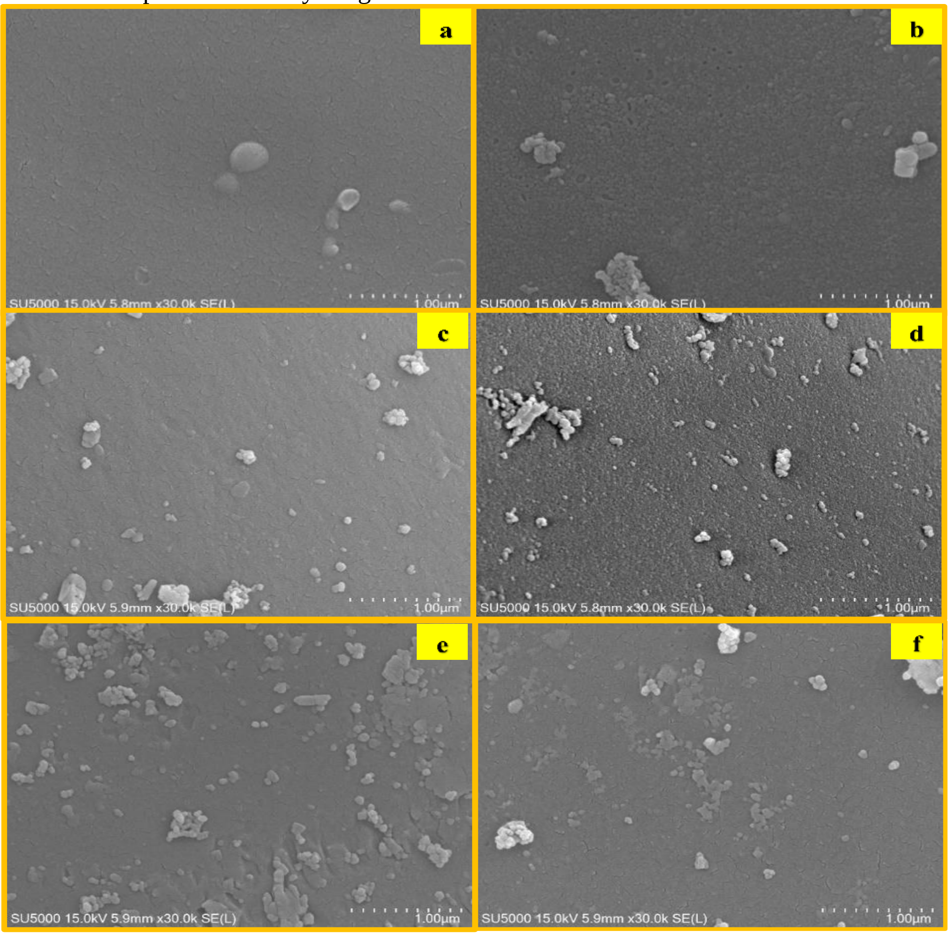

Figure 4: SEM images of the representative Ag-NPs/CWF samples for 60:40:7.5 in (a) and (b), 60:40:15 in (c) and (d), and 60:40:30 in (e) and (f) sintered at 850 °C and for different conditions of samples with presence and absence of Ag nanoparticles.

Silver ions must be released into the water to be effective as bactericidal agents. They must be attached to bacteria cells or directly transferred from a bulk silver source to bacteria cells. To sustain antibacterial activity, silver ions from the ceramic structure are slowly released into the water through mesopores. This presents similar results to one reported by the study of Mikelonis et al. (2020) and Oyanedel-Craver & Smith (2008). The nanoparticles formed (16–25 nm) were likewise demonstrated through the same analysis supporting the previous results. Among the three ratios studied, 60:40:7.5, 60:40:15, and 60:40:30, the results by ratio 60:40:7.5 sintered at 850 °C in Figure 4 showed the most excellent performance results. These results are therefore in reasonable agreement with the XRD results (Figure 3).

Table 2 shows the average analysis results of synthesized systems toward *E. coli* removal. It can be seen that the synthesized systems are capable of removing pathogenic *E. coli* and water contaminants were corrected by all the tested systems, especially the prepared system 60:40:7.5. The efficiency of the prepared ceramic powder filter samples was determined through the equation, $E = \left(C_0 - \frac{C_f}{C_0}\right) x\ 100$. Where C_0 is the initial concentration of a given parameter and C_f is the final concentration of the given parameter in the water samples.

Table 2: The table for water analysis results of *E. coli* from Ruaha River (Kalenga, Iringa), for the control sample and for Ag-NPs/CWF samples heated at 600 °C for Ag^+ reduction

Sample ID	***E. coli* (cfu/100mL)**
Control sample Before	162.0
60:40:30 (850 °C)	37.00
60:40:30 (650 °C)	124.0
60:40:7.5 (850 °C)	0.000
60:40:7.5 (650 °C)	17.33
60:40:15 (650 °C)	72.67
60:40:15 (850 °C)	40.67

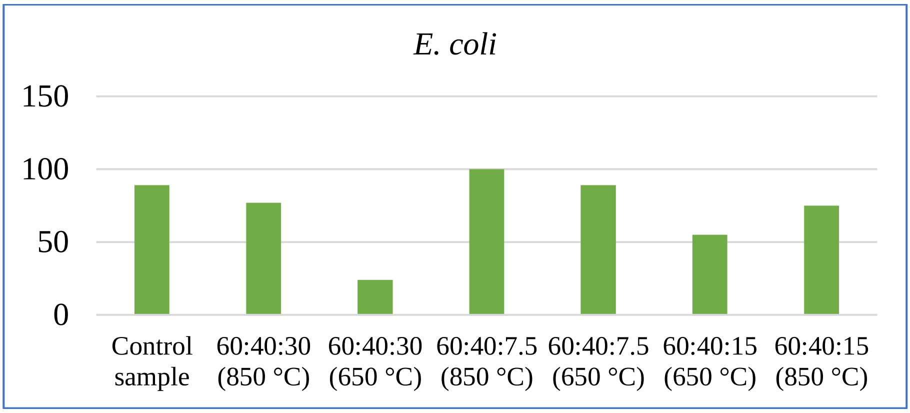

Figure 5: The efficiency percentage of synthesized systems on the water for *E. coli* correction.

Based on Figure 5, the calculated efficiency of the synthesized systems is shown to be effective in achieving the desired parameter corrections. The calculated efficiency of the tested synthesized systems provided a standard efficacy level of 99% for the best-selected ratio for *E. coli* removal from water. This was highly influenced by two factors, namely the ratio (amount) of the materials and the sintering temperature employed. In the literature, the higher the sintering temperature, the greater the influence on properties like porosity, structure, crystallinity, and metal reduction (Li et al. 2020). However, the ratio (amount) of the material employed in the synthesis process also plays a significant role in the system's efficiency. Therefore, it has been noted that the lower the amount the more excellent the performance of the resulting ceramic system. Reflect Figure 5 presenting the best performance in *E. coli* removal from the tested water, it is vivid considering the 60:40:7.5 system, which was sintered at 850 °C. In addition, the higher the ratio with low temperature, and the less efficient the system, as in the 60:40:30 systems, which was sintered at 650 °C. Similarly, Oyanedel-Craver and Smith (2008) reported *E. coli* removal rates ranging from 97.8% to 100%. Furthermore, their study suggested that factors influencing the release of silver into the filtered water encompass the quantity of silver integrated into the ceramics, the water's chemistry, and the pore structure of the ceramic material.

Conclusions

The sample synthesized using the ratio of 60:40:7.5 was observed to be the most effective for the removal of microbial water contaminants in comparison with the other two ratios (60:40:15 and 60:40:30). It was observed that (>99%) of *E. coli* bacteria can be eliminated from water using the ceramic filters. To improve water purification efficiency, it has been found that the sintering temperature significantly contributed to the formation of a well-structured and porous system, which improved the system's morphology and its efficiency. The analysis indicated the presence of reduced silver (Ag^0) is suitable for removing pathogenic microorganisms, such as *E. coli*, from contaminated water. The filter samples synthesized in this study were in powder form hence leading to easy employment, fabrication, less costly, and flexible to use. Nevertheless, because of their shape, size, and durability, ceramic filter samples in powdered form are portable. The synthesized water filter is simple to use because the ceramic powdered samples can be molded and placed in a container. It is possible to scale up the production of good quality ceramic water filters since they use locally sources and the preparation method is facile.

Competing interest

The authors declare that they have no competing interests

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Authors' Contributions

GMS participated in designing, writing, and submitting the manuscript. EE was a major contributor to the manuscript's writing and interpretation of the relevant literature. CF was a major contributor to the manuscript's writing and interpretation of the relevant literature. TEK was a major contributor to the manuscript's writing and interpretation of the relevant literature. GNS Origination of the water purification system idea, and conducted research relating to the chemical contaminants in water and final approval of the version to be submitted. All authors proofread the work before submission.

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