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Optimization of operational parameters for the treatment of roof-harvested rainwater with biologically synthesised nanosilver coated on sand

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ABSTRACT

Rainwater is a major source of drinking water in developing countries. Roof-harvested rainwater is generally microbiologically contaminated and thus needs to be treated effectively to meet drinking standards. Filtration of rainwater with sand coated by silver nanoparticles enhances the microbial removal efficiency. In this study, the filtration parameters of treating rainwater with biologically synthesized nanosilver coated sand are optimized. Of the various synthesis methods, the biological method was chosen due to benefits such as cost-effectiveness and its eco-friendly nature. Silver nanoparticles were synthesised using papaya fruit extract and then coated on sand. The synthesized nanosilver coated sand was subjected to characterization methods such as energy dispersive spectroscopy and X-ray diffraction (XRD) analysis. With silver coated sand as control, multiple long duration tests were performed to treat rainwater with nanosilver coated sand to find the optimal values for filtration parameters such as filter bed depth and empty bed contact time (EBCT). The crystallite size of the nanosilver coated sand was found to be 43.8 nm. The optimal values for filter bed depth and EBCT were found to be 12 cm and 15 minutes respectively. The rainwater treated with nanosilver coated sand met drinking water standard IS 10500: 2012 until the media got exhausted.

Key words: biosynthesis of nanosilver coated sand, energy dispersive spectroscopy, optimization of water filtration, roof-harvested rainwater treatment, X-ray diffraction analysis

HIGHLIGHTS

- Application of nanotechnology in water treatment.
- Eco-friendly biological synthesis of silver nanoparticles.
- Filtration of roof harvested rainwater using nanosilver coated sand.
- Long duration column tests to evaluate bacterial removal efficiency.
- Optimization of operational parameters (Bed depth & EBCT) of filtration process.

1. INTRODUCTION

Rainwater is one of the primary sources of potable water on the planet earth. Rainwater harvesting is adopted in various parts of the world, more prominently where conventional water supply systems cannot meet the water demands in the region. Rainwater harvesting is a method of collecting and storing rainwater using various techniques such as pots, tanks, cisterns, ponds or dams (Awawdeh *et al.* 2012). In developing countries where rainfall is available, there is a positive trend for harvesting rainwater on roof tops and using it for drinking purposes. However, studies have reported that harvested rainwater often does not meet microbiological drinking water quality standards since most of the contaminants found are faecal coliforms of animal origin (Shaheed *et al.* 2017). Also, various research confirms that roof harvested rainwater could be contaminated with various indicator and pathogenic organisms. Clearly, the need for an efficient and effective treatment of rainwater before using it for drinking purposes is well recognized (Lye 2002; Meera & Ahammed 2006; Awawdeh *et al.* 2012; Samuel & Mathew 2015; Shaheed *et al.* 2017).

Sand filtration is one of the commonly used techniques for treating the harvested rainwater. It is reported that sand coated with metal oxide/hydroxide is capable of removing various contaminants including bacteria from water (Meera & Ahammed 2006). The contaminant species are recalcitrant under normal geo-environmental conditions and natural attenuation to safe levels is not very plausible. The synthesis of particles that can counteract contaminants at nanoscale level (1–100 nm) has emerged as an effective remedy in water treatment applications. The technology refers to the study and manipulation of chemical and physical changes that occur at nanoscale level. Nanotechnology is used for developing new materials and devices in a variety of industries and it is also applied in water purification (Saravanan *et al.* 2020).

Sand coated with metal nanoparticles could be efficiently utilized for water treatment applications. Because of their smaller structure and distinctive crystallographic nature, nanoparticles have high surface to volume ratio and thus possess enhanced chemical activity. The antimicrobial capability of metal based nanoparticles, especially silver, has prompted their applications in the field of water purification (Gopinath *et al.* 2013; Kulkarni & Muddapur 2014; Aguirre *et al.* 2020). Various studies have shown that silver nanoparticles (AgNP) are an efficient medium with antibacterial activity towards many bacterial strains and microorganisms commonly present in medical and industrial processes (Saravanan *et al.* 2020).

Silver nanoparticles are synthesized traditionally by physical and chemical methods. However, these methods of synthesis are more expensive and energy consuming. Also, the chemical and physical methods of nanosilver production involve the use of toxic and hazardous chemicals, which may pose potential environmental and biological risks. Since the synthesized nanosilver coated sand has to be handled by humans, there is a need for a safe, eco-friendly and economical method of synthesis. The biological method is a cost-effective and eco-friendly approach, devoid of toxic chemicals and requires less use of energy. In the biological method of synthesis, the nanoparticles are obtained by utilizing bacteria, fungi, and plant materials. The synthesis of nanoparticles with plant extracts is comparatively more economical and is being used widely (Prabhu & Poulose 2012; Prasad 2014; Vijayaraghavan & Ashok Kumar 2017; Anjali Das *et al.* 2020; Mani *et al.* 2020).

Filtration is a widely accepted technique for the process of water treatment. Filtration is defined as the process of separating suspended and colloidal particles that are present in an aqueous solution by draining it through a porous medium. Filtration is a remedy for the common challenges in water treatment such as colour, turbidity and microorganisms. The factors influencing the filtration process can be categorized into suspension characteristics (type, size, density, hardness, water temperature, and suspended particles concentration), filter medium characteristics (type, granulometry, filter material-specific weight, and filter bed depth) and hydraulic characteristics (filtration rate, available hydraulic load, and effluent quality) (García-Ávila *et al.* 2020). In order to improve the water treatment process, the key filtration parameters such as filter medium characteristics (filter bed depth) and hydraulic characteristics (filtration rate in terms of empty bed contact time – EBCT) have to be optimized.

The main purpose of this study is to improve the process of treating roof-harvested rainwater with biologically synthesized nanosilver coated sand. This study focuses on enhancing the bacterial removal efficiency of nanosilver coated sand (biologically synthesized with papaya fruit extract) by optimizing the filtration parameters such as filter bed depth and EBCT. Accordingly, the objectives of this study were:

- to synthesis silver nanoparticles biologically using papaya fruit extract and subsequently coat them on sand for the treatment of rainwater harvested on rooftops. Also, to characterize the nanosilver coated sand.
- to carry out multiple long duration column tests in order to optimize the bacterial removal efficiency of nanosilver coated sand in treating rainwater. The long duration column tests are to be performed by keeping silver coated sand as control and systematically varying the parameters such as bed depth and EBCT.

2. MATERIALS AND METHODS

2.1. Rainwater samples and materials for filtration media

The rain water was collected from the concrete roof top of Mammiyoor residents in the Iringapuram village (10° 36′ 16″N & 76° 02′ 03″E) of Thrissur. The map of the location of the study is shown in Figure 1. The rainwater from the concrete roof (area of 20 m^2 and age of around 24 years) was collected in a Poly Vinyl Chloride (PVC) storage tank (age 1 year) with a capacity of 1,000 liters. The samples of rainwater for the study were collected from this storage tank using clean and dry plastic containers of 20 liters capacity. The collected samples were stored in plastic containers at low temperature (<100 °C) in a refrigerator.

River sand passed through a 850 μm sieve and retained on a 300 μm sieve was used to carry out this study. The chemicals used for this study were provided by Merck Millipore and were supplied to the study area by Chemind, Thrissur, Kerala, India. The chemicals used include silver nitrate ($AgNO_3$), 0.17 M pure sodium hydroxide (NaOH) and ammonium hydroxide (NH_4OH). Also, 9% sugar solution was used for the reduction process. All the chemicals used in this study were of analytical grade.

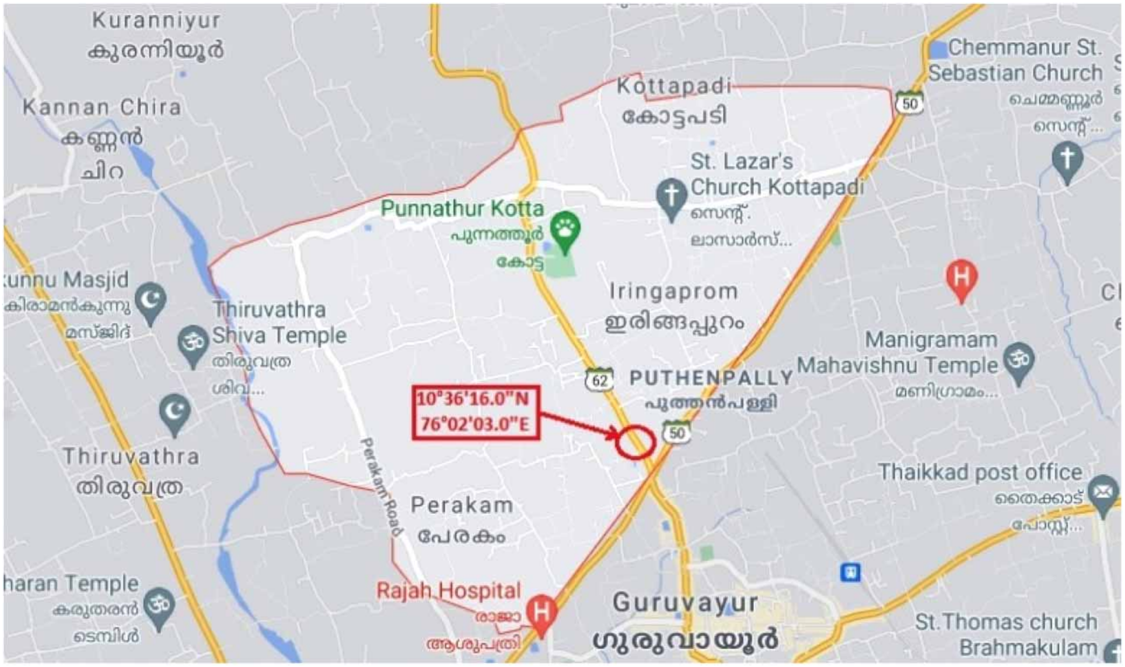

Figure 1 | Map showing the location of the study.

In this study, papaya fruit extract was used for the biosynthesis of silver nanoparticles. The papaya fruit extract was used because of its inherent anti-microbial properties and its local availability. The papaya fruit extract was prepared according to the method reported by Sreejamol *et al.* (2014).

2.2. Preparation of filtration media

Firstly, the silver was coated on sand as per the method reported by Mahmood *et al.* (2008). 500 gm of graded, washed and dried sand and $AgNO_3$ (1 mM) were mixed and then dissolved in one litre of distilled water. The components were thoroughly mixed and then allowed to mature for one hour. Subsequently, the mixture was treated with 0.17 M pure NaOH and thoroughly mixed again. The sand was then treated with 1:1 NH_4OH solution and 9% sugar solution (reduction) in the respective order. Between each addition, the components were mixed thoroughly and then left for one hour. The treated sand was solar dried and washed subsequently with distilled water to pH 7. The sand was then oven dried again at 100–110 °C.

The nanosilver coated sand was prepared by treating the silver coated sand with the papaya fruit extract. The papaya extract and silver coated sand were mixed in 1:9 ratio in order to form silver nanoparticles by reduction of silver. The mixture was left at room temperature for about 5 hours. It was finally washed and oven dried to remove the unwanted particles. The silver nanoparticles coated on sand forms an efficient filter media for treating rainwater. By coating the silver nanoparticles on sand, activity loss due to agglomeration is prevented. Also, coating of silver nanoparticles on sand facilitates the easy separation and reduction of excessive pressure drops in the case of flow-through systems.

2.3. Characterization of nanosilver coated sand

The characterization of the filter media was performed using energy dispersive spectroscopy (EDS) in order to quantify the weight percentage of silver coated on the sand. Both the nanosilver coated sand and the silver coated sand were characterized by EDS. Furthermore, the nanosilver coated sand was also characterized using X-ray diffraction (XRD) analysis.

The EDS characterization of the filter media was carried out in the Centre for Microscopy, National Institute of Technology, Calicut, Kerala, India. The EDS analysis was performed using Energy Dispersive Spectroscopy (Horiba, Japan). The XRD analysis was performed in the Centre for Materials for Electronics Technology, Athani, Thrissur, Kerala, India using a D5005 diffractometer (Bruker, Germany).

The characterization by XRD revealed the crystallite size and shape of the synthesised particles. XRD analysis was performed using a diffractometer in the 2θ range of 20–60°. The crystallite size of nanosilver coated sand was calculated using Debye-Scherer's formula:

$$D = \frac{K\lambda}{\beta \cos \theta} \tag{1}$$

where D is the crystal size of nanosilver coated sand, K is the constant of the Debye-Scherer equation, λ is the wavelength of the X-ray source, β is the full width at half maximum of the diffraction peak (half of the distance between two extremes of the peak) and θ is the Bragg angle (mid-point value of two extremes of the peak). Also, the interplanar distance (d) can be calculated using Bragg's law:

$$\lambda = 2d sin\theta \tag{2}$$

2.4. Roof-harvested rainwater samples

After bringing it to room temperature and mixing thoroughly, the required amount of rainwater samples were taken daily from the stored containers. By performing the standard methods (APHA, 2017), the characteristics of the collected samples such as pH, turbidity, hardness, total dissolved solids, silver and faecal coliform were determined. The multiple tube fermentation technique was carried out as needed to determine the faecal coliform count – the key parameter of the study. The concentration of faecal coliform was found to be lower in the collected rain water. Hence, cow dung was added to the sample rainwater to increase the influent bacterial concentration: 2 ml of cow dung slurry (1 g of cow dung in 10 ml of distilled water) was added to 20 liters rainwater. Also, by adding cow dung, almost the same number of coliforms was ensured in the water samples so that comparison of efficiency by adjusting filter media parameters could be made. The characteristics of cow dung spiked rainwater were also found.

2.5. Experimental setup for long duration column test

The schematic diagram of the experimental setup for the long duration column tests is shown in Figure 2. Two glass columns of 7.3 cm diameter were setup to carry out the long duration tests with nanosilver coated sand and silver coated sand. The silver coated sand acted as control throughout the long duration tests. The column tests were performed to evaluate the efficiency of nanosilver coated sand in comparison to that of silver coated sand in removing faecal coliforms from the cow dung spiked rainwater samples. A perforated plate was used at the bottom of each column in order to prevent media loss. Before

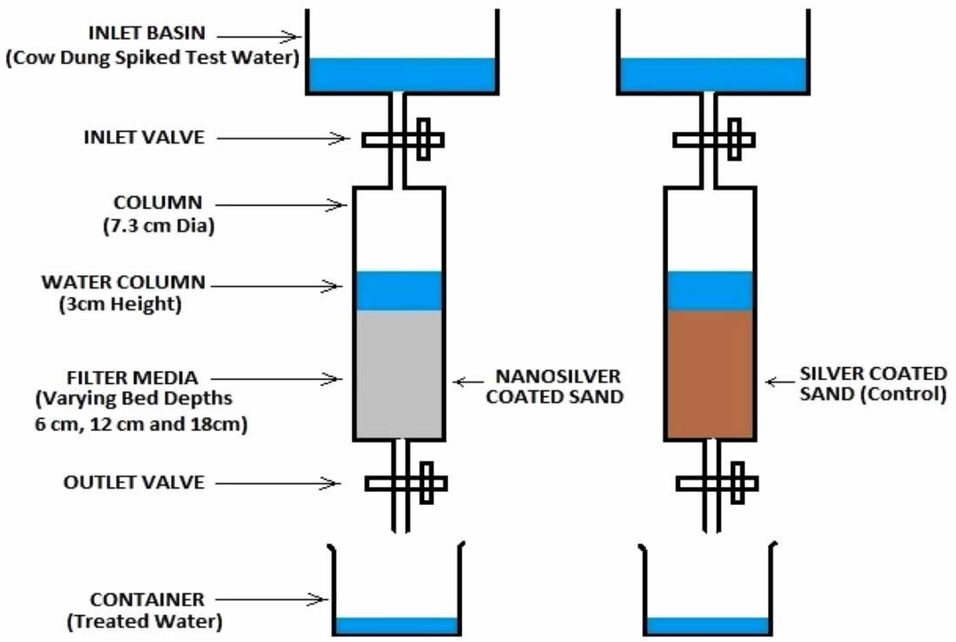

Figure 2 | Set up for long duration column tests with nanosilver coated sand and silver coated sand (control).

carrying out the filtration of bacteria spiked roof-harvested rainwater, de-ionized water was passed through the columns till the unbound coating was removed and the column effluents were free of precipitates. The initial faecal coliform count of the cow dung spiked rainwater was found and the down flow long duration column tests were performed with EBCT of 15 minutes and varying filter bed depths of 6, 12 and 18 cm. EBCT is a measure of the time for water in contact with the filter media assuming that all water passes through at the same velocity. The inlet and outlet valves were adjusted to control the flow rate and maintain the required EBCT.

A constant head of 3 cm water was ensured over the media throughout the long duration column tests. The tests were continued until break-through of the media occurred. The effluent samples were analysed for faecal coliforms at a regular time interval of 6 hours. The long duration tests were carried out with filter media (nanosilver coated sand and silver coated sand) packed to bed depths of 6, 12 and 18 cm respectively with an EBCT of 15 minutes. The optimal bed depth of the nanosilver coated sand in removing bacteria from the test water was found from these long duration column tests. Finally, by packing the filter media to the optimal bed depth, the long duration column test was again performed with an EBCT of 30 minutes.

3. RESULTS AND DISCUSSION

3.1. Characteristics of influent water

The influent characteristics of rainwater and cow dung spiked rainwater used for the study are given in Tables 1 and 2 respectively.

3.2. Characterization results

EDS was used to quantify the weight percentage of silver on different filter media.

3.2.1. Nanosilver coated sand synthesised with papaya extract

Figure 3 and Table 3 show the energy dispersive spectrum and elemental composition of nanosilver coated sand synthesised using papaya fruit extract, respectively.

The EDS analysis and the elemental composition confirm the presence of silver in the nanosilver coated sand. Also, it is reported that the size of nanoparticle synthesised using this biological method with papaya fruit extract comes under 100 nm (Sreejamol *et al.* 2014). The XRD spectrum of nanosilver coated sand synthesised with papaya fruit extract is shown in Figure 4.

Table 1 | Influent characteristics of roof-harvested rainwater

Parameter	Unit	Value
Faecal coliform	MPN/100 ml	40
pH	–	6.59
Hardness	mg/l	55
TDS	mg/l	48.1
Turbidity	NTU	4

Table 2 | Influent characteristics of cow dung spiked rainwater

Parameter	Unit	Value
Faecal coliform	MPN/100 ml	1.1×10^9
pH	–	6.74
Hardness	mg/l	52
TDS	mg/l	52.6
Turbidity	NTU	10
Silver	mg/l	Below Detection Level

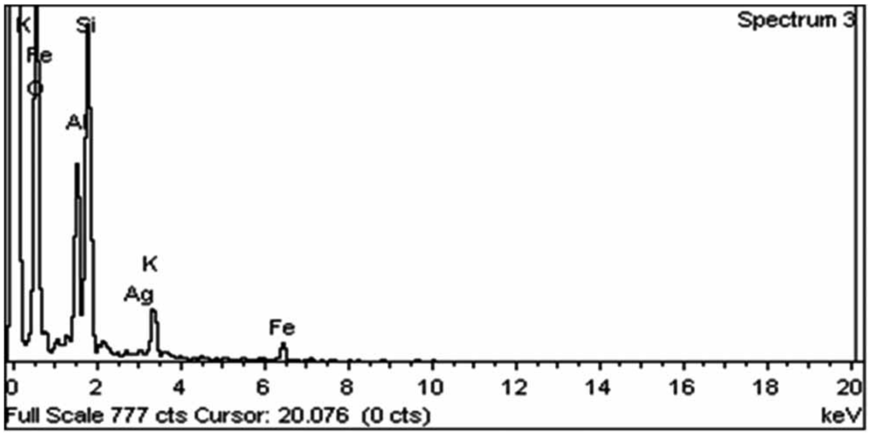

Figure 3 | Energy dispersive spectrum of nanosilver coated sand.

Table 3 | Elemental composition of nanosilver coated sand

Element	Weight %	Atomic %
O K	57.91	73.07
Al K	10.35	7.74
Si K	20.17	14.50
K K	4.82	2.49
Fe K	5.39	1.95
Ag L	1.35	0.25
Total	100.00	

The crystallite size of nanosilver coated sand was calculated using Debye-Scherer's formula. With the values $\beta = 0.187°$, $K = 0.9$ and $2\theta = 28.162°$, the crystallite size of nanosilver coated sand was found to be 43.8 nm. The interplanar distance was calculated using Bragg's law and was estimated to be 0.3169 nm.

3.2.2. Silver coated sand

The energy dispersive spectrum of silver coated sand is shown in Figure 5.

Table 4 shows the elemental composition of silver coated sand.

3.3. Long duration column tests

Long duration column tests were performed with cow dung spiked rainwater by filtering through nanosilver coated sand and silver coated sand by varying the parameters of filtration. The results of the column tests are given below.

3.3.1. Variation of filtration bed depth

The log unit removal of faecal coliform by nanosilver coated sand and silver coated sand from rainwater samples for filtration bed depths of 6, 12 and 18 cm are shown in Figures 6–8 respectively. EBCT of 15 minutes was maintained. The effluent samples were collected every 6 hours and analysed for bacterial removal until the media got exhausted.

The effluent quality deteriorated at 24 hours with the filter bed depth of 6 cm, while the same occurred at 36 hours in the case of 12 cm bed depth. However, when the bed depth was increased to 18 cm, the effluent quality deteriorated again at 36 hours. Hence, long duration column tests were not conducted further with higher filter bed depths. The variation of breakthrough time for nanosilver coated sand with different bed depth values is shown in Figure 9. The optimal bed depth for filtration was found to be 12 cm.

Bacteriological treatment efficiency of water with sand as filter medium is not sensitive to high filter bed depths. It is reported that the optimum filter bed depth for treating water with sand to produce satisfactory water quality is 40 cm. The

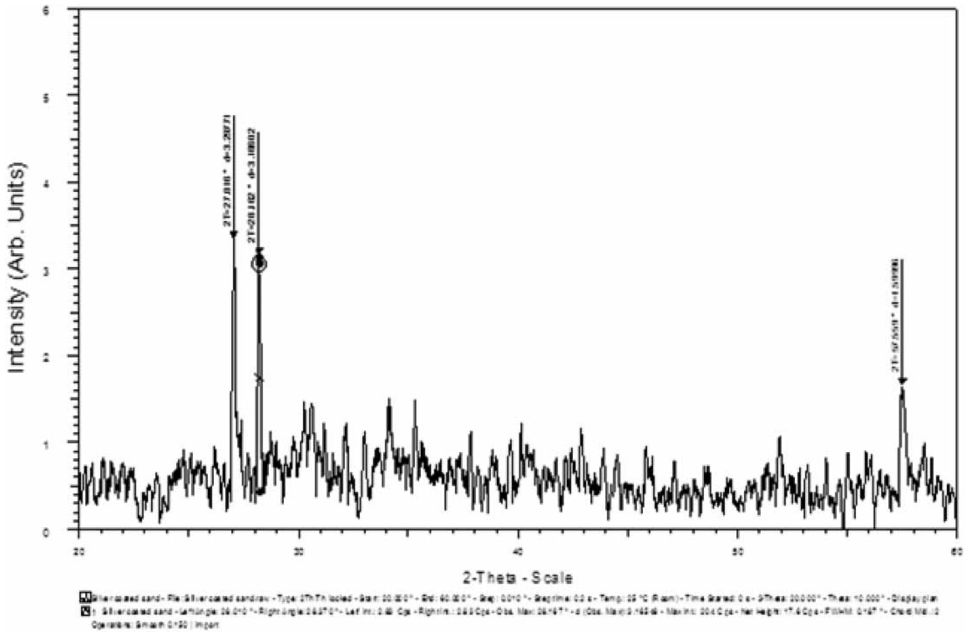

Figure 4 | XRD spectrum of nanosilver coated sand.

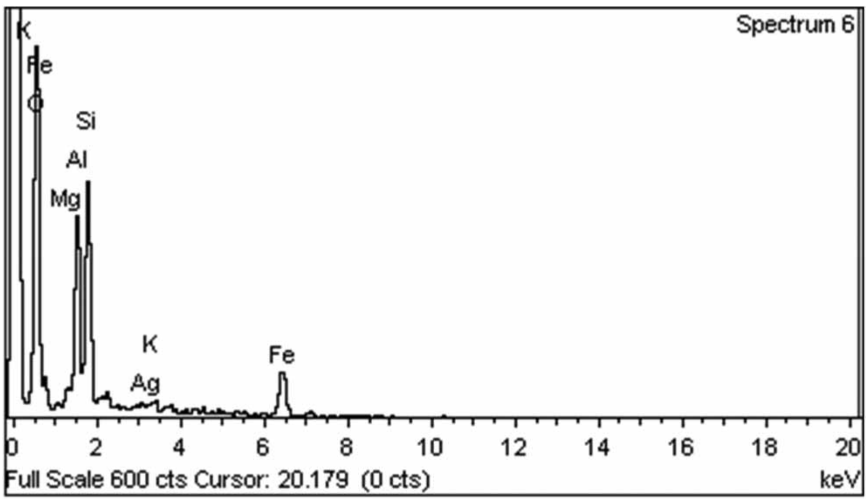

Figure 5 | Energy Dispersive Spectrum of Silver Coated Sand.

Table 4 | Elemental composition of silver coated sand

Element	Weight %	Atomic %
O K	51.97	69.78
Mg K	1.37	1.21
Al K	12.03	9.58
Si K	15.83	12.11
K K	0.99	0.54
Fe K	17.41	6.70
Ag L	0.39	0.08
Total	100.00	

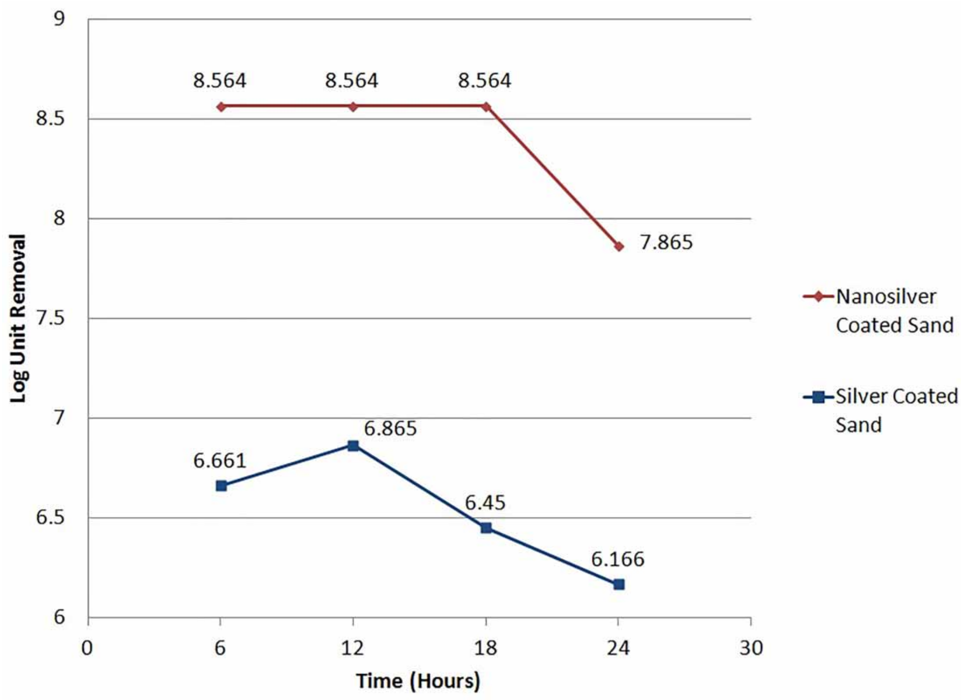

Figure 6 | Log unit removal of faecal coliform against time for 6 cm bed depth.

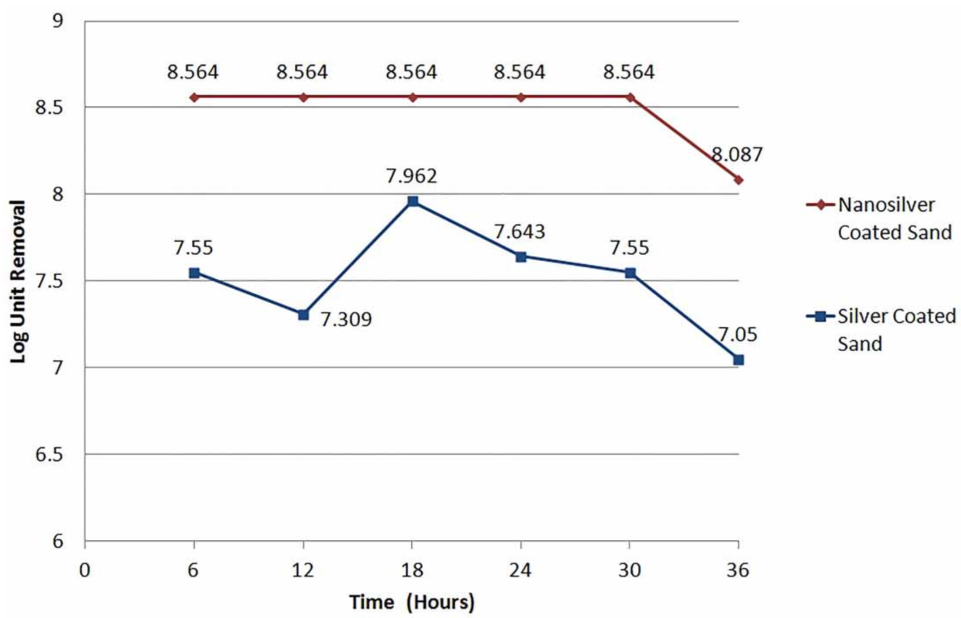

Figure 7 | Log unit removal of faecal coliform against time for 12 cm bed depth.

bacterial purification of water occurs within the top 40 cm of the filter bed (Muhammad *et al.* 1996). Comparatively, in this study, the optimal bed depth for treating rainwater with nanosilver coated sand to meet the drinking water standards is only 12 cm. The reduced filter bed depth is due to the enhanced capability of nanoparticles in removing microbes from rainwater. Also, the antimicrobial capability of nanosilver coated sand can be further improved by increasing the amount of silver nanoparticles coated on the sand. More studies are needed to explore various methods of efficiently coating an increased amount of silver nanoparticles on sand.

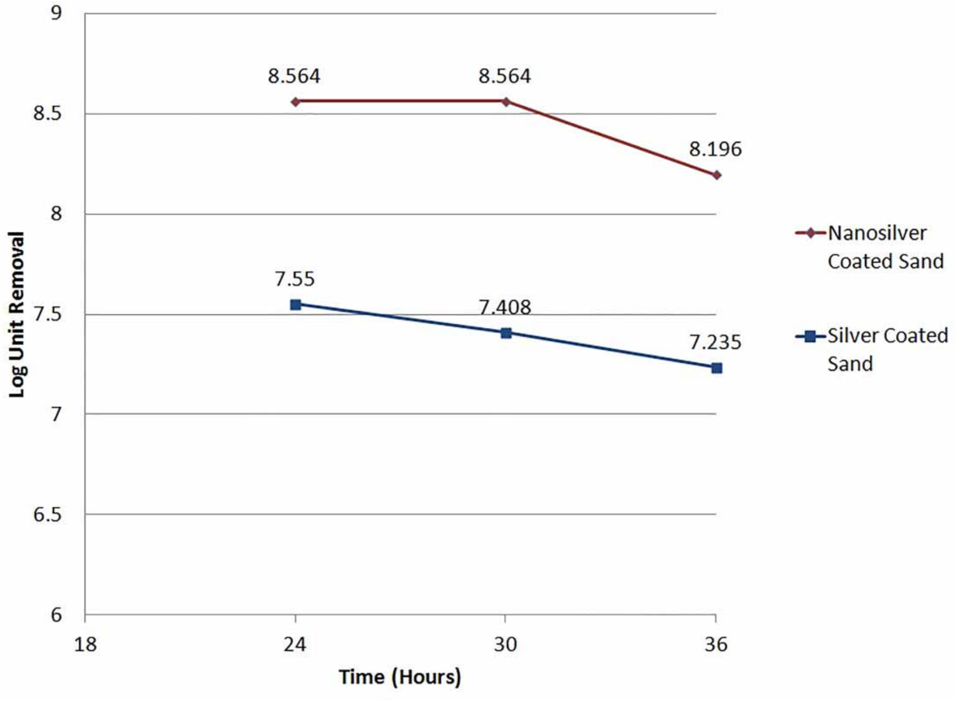

Figure 8 | Log unit removal of faecal coliform against time for 18 cm bed depth.

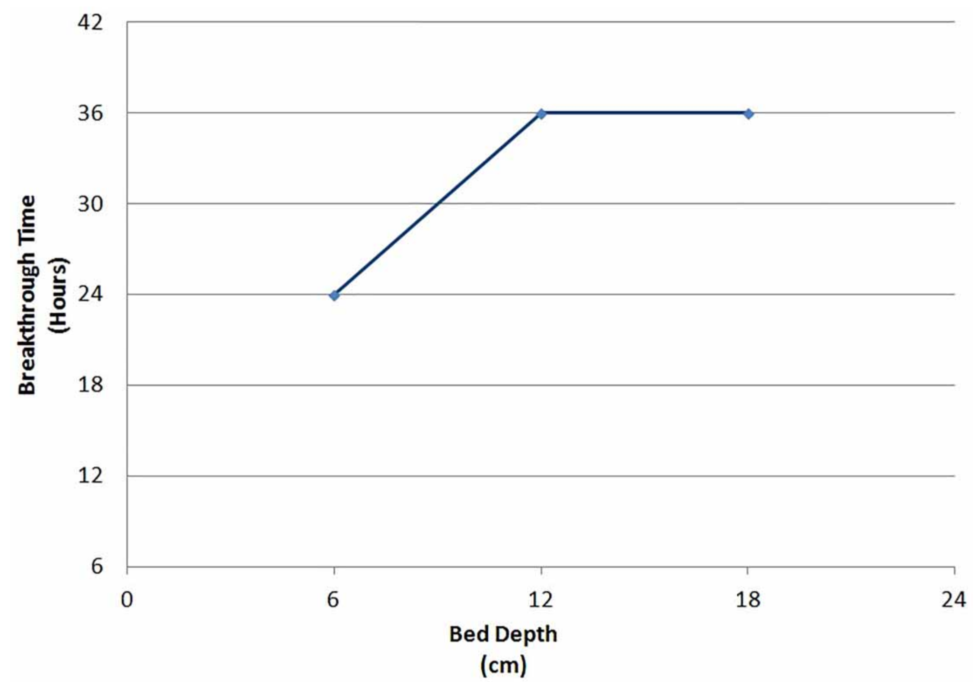

Figure 9 | Breakthrough Time against Bed Depth for Nanosilver Coated Sand.

3.3.2. Variation of empty bed contact time

The long duration column test was also conducted to analyse the effect of EBCT on the efficiency of removing coliforms from roof harvested rainwater. By keeping the filter bed depth to the optimal value of 12 cm, the column test was again performed

Table 5 | Effluent characteristics of treated water

Parameter	Unit	Silver coated sand	Nanosilver coated sand
Faecal coliforms	MPN/100 ml	31	<3
pH	–	6.31	6.74
Hardness	mg/l	48	52
TDS	mg/l	53.7	58.1
Turbidity	NTU	2	1
Silver	mg/l	BDL	0.05

with an EBCT of 30 minutes. With the filter bed depth of 12 cm and EBCT of 30 minutes, the quality deterioration of the effluent also occurred at 36 hours. The log unit removal of faecal coliform was found to be 7.59, which was not significantly different from the value obtained with bed depth of 12 cm and EBCT of 15 minutes. Hence, the optimal value for EBCT in treating rainwater samples with nanosilver coated for a 12 cm bed depth was fixed as 15 minutes.

3.4. Effluent characteristics

The effluent characteristics of harvested rainwater treated by nanosilver coated sand and silver coated at the end of 30 hours (exhaustion time) were also recorded. The effluent characteristics of treated water are shown in Table 5.

The various effluent parameters of the rainwater treated with nanosilver coated sand met drinking water standards as per IS 10500:2012. However, for the water treated with silver coated sand, parameters such as pH and faecal coliforms did not meet the drinking water standards. The faecal coliforms in the sample rainwater are removed by various processes such as sedimentation, straining, adsorption, and chemical action. The antimicrobial activity of biologically synthesized nanosilver coated sand enhanced the removal of faecal coliform from the rain water samples. Also, previous studies have reported that particle sizes of silver ranging between 1 and 100 nm have an effect on the antibacterial properties of nanoparticles. Moreover, silver nanoparticles cause irreversible damage to the cellular membrane, which enables the accumulation of nanoparticles in the cytoplasm. The antimicrobial action of silver nanoparticles is due to this damage and not because of toxicity (Monyatsi *et al.* 2012).

4. CONCLUSION

The bacterial removal efficiency of nanosilver coated sand in comparison to silver coated sand was analyzed in this study. Compared to the traditional physical and chemical synthesis of silver nanoparticles, biological synthesis has advantages such as cost-effectiveness and its eco-friendly approach. Silver nanoparticles were biologically synthesized with papaya fruit extract and their characterization was performed using EDS and XRD analysis methods. The elemental composition of the filter media was found from the EDS analysis and it confirmed the presence of silver. Also, the crystallite size of the nanoparticle was found to be 43.8 nm from the XRD analysis. The two important filtration parameters (filter bed depth and EBCT) were optimised for maximum faecal coliform removal efficiency from roof- harvested rainwater. The optimum values for filter bed depth and EBCT in treating rainwater with nanosilver coated sand were found to be 12 cm and 15 minutes, respectively. The study was conducted with elevated faecal coliform concentration that is generally found in highly polluted water. Roof-harvested rainwater is less microbiologically contaminated. Hence, it can be inferred that biologically synthesized nanosilver coated sand can be used as an effective filter medium for removing microbiological contamination from roof-harvested rainwater. Moreover, the process of treating rainwater with nanosilver coated sand can be improved by optimizing the filtration parameters such as filter bed depth and EBCT. In comparison to the treatment of water using sand having an optimum filter bed depth of 40 cm, the nanosilver coated sand was able to produce drinking water standards at a lower optimum bed depth of 12 cm. Furthermore, the antimicrobial property and life span of nanosilver coated sand as a filter media can be enhanced by coating a greater amount of silver nanoparticles on the sand. Hence, it would be beneficial to explore various methods for coating increased amounts of silver nanoparticles so that the water treatment process can be further improved.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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