

Article

Potable Water Treatment in a Batch Reactor Benefited by Combined Filtration and Catalytic Ozonation

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Abstract: Due to continuous contamination of groundwater by anthropogenic activities, potable water fetches numerous pollutants such as pathogens, pharmaceuticals, and heavy metals, with these being severe health hazards. The main aim of the current study was to develop a hybrid unit based on catalytic ozonation and the filtration process to effectively remove the contaminants in drinking water. To the best of our knowledge, in the current study, the Fe-Zeolite 4A (Fe-Z4A)/O₃ process followed by filtration involving rice husk and activated carbons were studied for the first time in order to treat drinking water. In the current investigation, fecal coliforms, arsenic, pharmaceuticals, turbidity, and TDS removal were investigated in a novel hybrid reactor. The results showed 100%, 45%, 40%, 70%, and 95% fecal coliform, arsenic, TDS, paracetamol, and turbidity removal efficiency, respectively. The results further indicated that all the studied drinking water samples followed WHO guidelines and NEQS for drinking water quality after the proposed treatment. Therefore, it is concluded that the proposed hybrid process implies a single unit is highly efficient for drinking water treatment. The designed novel hybrid reactor treatment can be scaled up in the future for household or commercial use.

Keywords: catalytic ozonation; water treatment; zeolites; activated carbon

1. Introduction

Water covers more than 70% of the Earth's surface, but only 0.01% of the available freshwater is potable; the remainder is in the form of glaciers and ice [1]. Groundwater is the major source of potable water all around the world. Water is considered safe and healthy when there is no substantial health risk associated with its consumption [2]. For the developed countries, the situation is not alarming as clean drinking water is accessible to 95% of the population and adequate disposal facilities are available for about 90% of the population [3]. According to the United Nations, at least 2.5 billion people in developing countries lack adequate sanitary systems, and over 780 million people lack access to clean drinking water, which is essential for survival [4]. As a result, approximately 2.3 billion people were estimated to be suffering from water-related diseases around the world in

2017, and this figure is expected to be even higher now [5]. In Pakistan, only a few cities have water treatment facilities, but those that do exist are often not completely functional and reported microbial pollution [6]. Currently, in most parts of the country, drinking water has become a major problem, and people in these areas travel 2 to 10 km away from their homes to fetch water. In major cities, such as Lahore, water supply systems have contaminations by arsenic, fluoride, and microorganisms. So, under these conditions, it has become very difficult to have healthy safe water at the household level. The multi-barrier approach is recognized as the best approach to minimize the risks linked with drinking unhealthy water. The multi barrier approach can be implemented at both the household and community levels. Every society mostly uses this basic approach, which involves filtration and disinfection. As the world's population is growing day by day, freshwater demand is increasing, and the availability of safe drinking water has become a major challenge around the globe. Healthy drinkable water must be free from all pollutants such as pathogens, micropollutants, and organic matter [7].

Over the years, many treatment technologies have been tested for drinking water treatment. Among the conventional treatment technologies, adsorption [8,9], coagulation-flocculation [10], filtration [11], and disinfection by chlorination [12,13] were implied. These technologies have advantages and certain limitations [14,15]. The limitations such as disinfection by-products formation (chlorination), regeneration cost or lack of rapid removal of pollutants (adsorption), removal of selected pollutants on the basis of their size (filtration), sludge production, and pH dependency (coagulation/flocculation) demands the investigation of novel hybrid technologies that may be capable of removing multiple pollutants. Therefore, in recent years, many novel technologies such as a variety of membrane filtration processes [16], oxidation processes [14,17,18], and a combination of conventional and advanced treatment were investigated [19–21].

Among the various techniques, catalytic ozonation has gained much success in the last decade. Catalytic ozonation has shown a great advantage towards organics and has been proven to be highly efficient in water treatment [22]. In this process, a catalyst is used to speed up the decomposition of soluble ozone, resulting in highly reactive hydroxyl radicals. These non-selective radicals have a great ability to oxidize refractory and harmful organic compounds into less toxic inorganic materials. Natural zeolites are ion-exchange and sorption materials with exceptional properties. These are economically and environmentally acceptable hydrated aluminosilicate materials. Natural zeolites have a wide range of applications due to their unusual three-dimensional porous structure [23,24]. The efficacy of natural zeolites in various water treatment systems is determined by their physical-chemical properties, which are inextricably linked to their geological origins. As these have a negative charge on their surface, they are cationic exchangers, which have resulted in the isomorphic substitution of silicon by aluminum in primary structural units. Numerous tests have shown that zeolites are very effective at extracting metal cations from water and wastewater. Chen et al. [25] studied the catalytic ozonation of nitrobenzene ZSM5 zeolites laden with metallic oxides of different metals (Ce, Fe, or Mn). Total organic compounds (TOC) removal efficiencies with NaZSM5-38, HZSM5-38, and NaZSM5-100 were 45.9%, 62.3%, and 59.0%, respectively. In total, there was 39.2% removal by single ozonation. Ikhlaq et al. [26] wanted to highlight the potential of ozonation and its efficiency with ZSM-5 zeolites for removing common water contaminants such as pharmaceuticals, VOCs, and carboxylic acids, as well as ozonation by-products. For the attained efficiency mechanism, the effects of various involved factors such as pH; adsorption; and involvement of hydroxyl radical scavengers, phosphates, and humic acids have also been investigated.

In the current investigation, a detailed study was conducted to remove pathogens, TDS, pharmaceuticals, turbidity, and arsenic from drinking and challenging water in a single unit, implying a combination of conventional and advanced treatment technologies. Moreover, to the best of our knowledge, in this study, we suggested for the first time a combination of catalytic ozonation using zeolite-4A followed by filtration by suggesting rice husk and activated carbon in a single unit. In the current investigation, an effort was made

to practically apply the combination of treatment methods to develop a household-type water purification system. This study may bring about economical and environmental benefits and can help to solve water-related issues such as lack of availability of clean water, disease prevention, and quality of water. Moreover, the current study also helps to achieve UN sustainable development goals 3, 6, and 9.

The current study focuses on the development of a novel hybrid unit based on catalytic ozonation and the filtration process to effectively remove the contaminants in drinking water. In the current research, treatment of potable water in Lahore, Pakistan, was performed in a two-stage batch reactor. The first stage treatment was performed by Fe-Z4A catalytic ozonation, and the second stage treatment was through a multi-bed filtration process. In the filtration bed, the three layers consisted of gravel, rice husk, and activated carbon. The removal of pathogens, arsenic, pharmaceutical pollutants, TDS, and turbidity was studied in potable water to make it safe under health considerations.

2. Materials and Methods

The paracetamol and Zeolite 4A were obtained from Sigma Aldrich U.K. All the other chemicals and reagents such as HCl, NaOH, H₂SO₄, lactose, and EC broth were obtained from Merck Germany. All the chemicals implied in the current investigation were of analytical grade and were used without any further purification.

2.1. Analytical Methods

The potable water parameters such as pH, total dissolved solids (TDS), pathogens (fecal coliforms), and arsenic (As) were measured by “Standard methods for the examination of water and wastewater” [27]. The pH and turbidity of water were measured by Hanna HI-9811 and HACH 2100 P, respectively. The concentrations of arsenic and paracetamol in the water sample were estimated by an atomic adsorption spectrophotometer (AAS) with graphite furnace (PerkinElmer Analyst-800, Waltham, MA, USA) and high-performance liquid chromatography (HPLC), respectively. An ozone generator (DA-12025-B12, Pakistan) was used for the catalytic ozonation process, and the O₃ dose was calculated by the iodometry method [27].

2.2. Water Sampling

Water samples were taken from four [4] different locations in eastern Lahore. The sample sites were selected as

1. Lahore press club housing scheme, Harbanpura (T1);
2. Nazir garden Lahore (T2);
3. Mughalpura Lahore (T3);
4. Taj Bagh Lahore (T4).

All four samples were collected from the WASA water supply. To certify the statistical significance, three samples were collected from each area, making a total of 12 samples. After collecting these samples, they were shifted to the laboratory and stored at 4° temperature. Figure 1 indicates the studied area.

2.3. Initial Characterization of Real Water

The initial characterization of real water is summarized in the Table 1.

Table 1. Initial characterization of water.

Parameter	Before Treatment	After Treatment	NEQS
pH	7 ± 0.3	7 ± 0.3	6.5–8.5
Pathogens	18 ± 8 MPN/100 mL	0 MPN/100 mL	0 MPN/100 mL
Arsenic	58 ppb	32 ppb	50 ppb
Turbidity	23 NTU	2.5 NTU	<5 NTU
TDS	448 Mg/L	329 Mg/L	<1000 mg/L

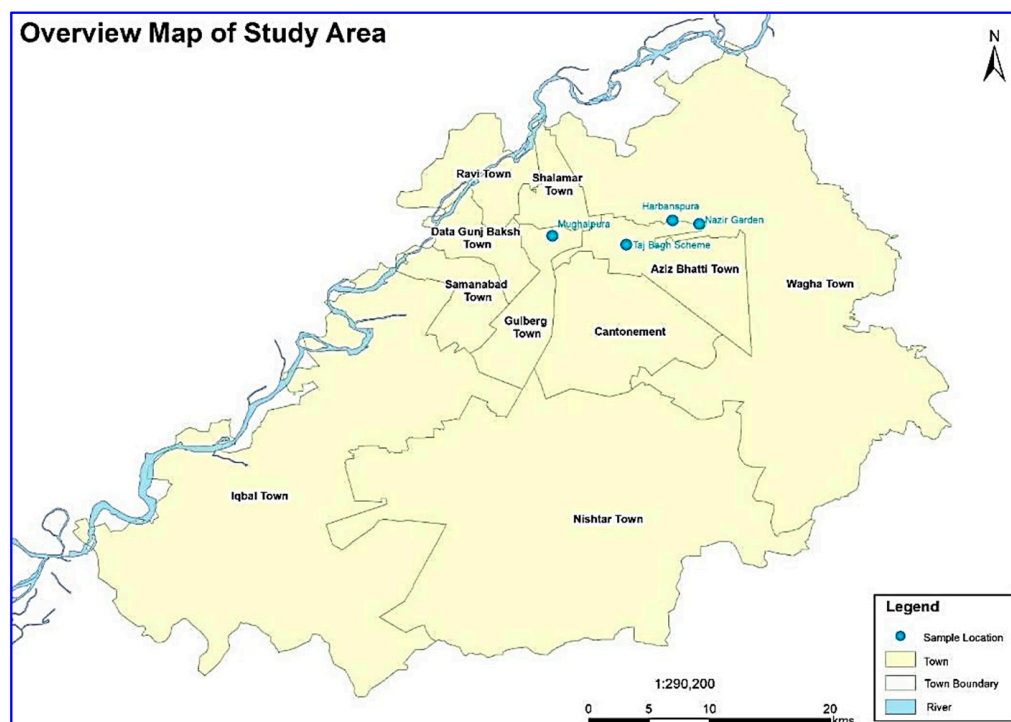


Figure 1. Overview of the study area.

2.4. Preparation of Challenging Water

In addition to the mentioned collected samples, challenging water of known concentration was also prepared in the laboratory to check the efficiency of zeolites and filters under study. Combined challenging water with contaminants such as TDS, pathogen, turbidity, and pharmaceuticals was prepared in the lab.

2.5. Experimental Setup

A semi-batch reactor was made from glass that was composed of two sections, each having a 19 L capacity and separated by a glass shield with the only passage for water to move being from the upper section to the lower. The top section consisted of the first stage treatment of water by catalytic ozonation. Z4A catalyst was suspended, and circulation was performed by a circulation pump. The lower section consisted of a multi-bed filter second-stage treatment. The multi-bed filter consisted of three beds consecutively of rice husk, gravel, and activated carbon. Water after stage 1 treatment was allowed to pass through second stage treatment. In each run, 19 L of the sample was taken in the reactor. Following the establishment of the optimal ozone dose, samples were taken for analysis at predetermined intervals. Figure 2 shows the experimental setup.

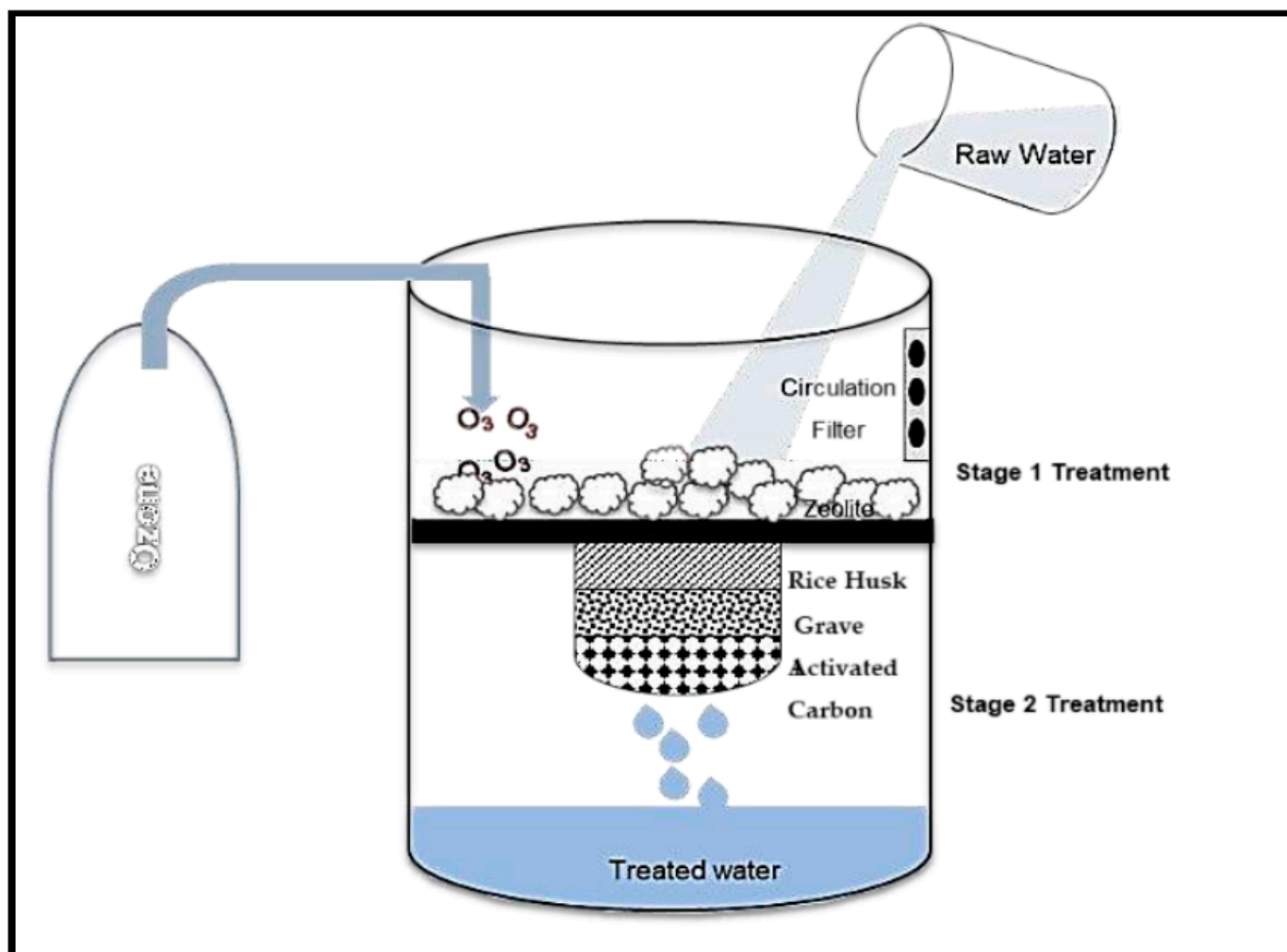


Figure 2. Cross-sectional view of the designed reactor.

3. Results and Discussion

3.1. Characterization of Catalyst

Table 2 summarizes the characterization of the catalyst. The surface area and pore size were studied by the BET method using a micromeritics USA ASAP analyzer. In this method, the nitrogen adsorption and desorption studies were conducted, and adsorption isotherms were implied by using nitrogen adsorption at 77 K; finally, surface area and porosities were determined by implying the kelvin equation and BJH method. The pore size of the catalyst was 4 angstrom (Å) or 0.4 nm, and the surface area was 90.55 m²/g. The thermal decomposition temperature of the catalyst was 700 °C. The point of zero charges was 6.5 ± 0.3 by using the mass titration method.

Table 2. Properties of Z4A catalyst.

Pore Size (Å)	4
Thermal decomposition (°C)	700
Surface area (m ² g ^{−1})	90.55
Point of zero charge (pH _{pzc})	6.5 ± 0.3
wt% iron (EDX)	8.5

3.2. Effect of Reaction Time

In this study, catalytic ozonation was performed to optimize its reaction time as stage 1 treatment in the upper section of the batch reactor and multi-bed filtration as stage 2 in the lower section for removal of desired pollutants. The removal of pathogens was studied by varying the reaction time of catalytic ozonation from 0 to 60 min, and samples were collected after every 10 min. For analysis, samples with 50 ± 8 MPN/100 mL values of pathogens and those prepared in the laboratory were used. Results (Figure 3) revealed that 100% of pathogens were removed at 30 min. On the basis of the results, 30 min was selected as the optimized reaction time for catalytic ozonation treatment.

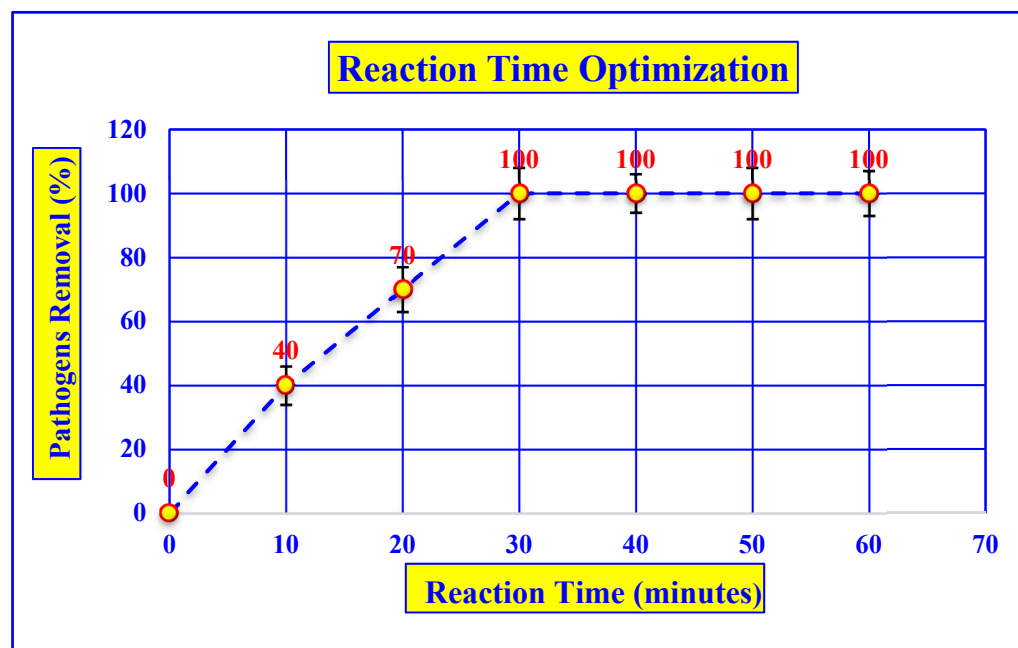


Figure 3. Reaction time optimization in catalytic ozonation (initial ozone flow, $O_3 = 0.9$ mg/min; off-gas ozone flow = 0.2 mg/min; O_3 consumption = 0.7 mg/min, pH = 7; T = 30 min).

3.3. Effect of Catalyst Dose

To analyze the effect of catalytic dose, several experiments were performed in the semi-batch reactor using different doses of catalyst varying from 5 to 20 g. Results (Figure 4) exhibited that as the value of catalyst dose was increased from 5 g to 20 g, reduction in bacteria also increased, but it became stable after 14 min with a maximum reduction of 100% pathogens using 20 g. In the case of a low catalyst dose (5 g), maximum efficiency was achieved after 28 min. With a 10 g dose, 100% removal efficiency was achieved in 24 min. It was also observed that with an increase in dose, the surface area also increased, which led to a reduction in bacteria. So, the results showed that with the increase in catalytic dose, an increase in removal efficiency was achieved. However, due to economical balance, 10 g was used as an optimum amount of catalyst dose.

In catalytic ozonation, active sites of Lewis and Bronsted acidities depend on the pH on the surface of zeolites. Bronsted acids are surface hydroxyl groups and ozone after reaction with these hydroxyl groups produced hydroxyl radicals. These hydroxyl radicals result in the disinfection of bacteria.

3.4. Pathogens Analysis

Samples collected from different areas were analyzed for the removal of pathogens by catalytic ozonation.

The results (Figure 5) show that after catalytic ozonation, all samples reported zero pathogens. The 100% reduction was achieved in 24 min with a catalytic dose of 10 g as

compared to simple ozonation in treatment time of 30 min. This could have been due to the production of higher hydroxyl groups as compared to simple ozonation. These hydroxyl groups inactivate the cell walls of pathogens.

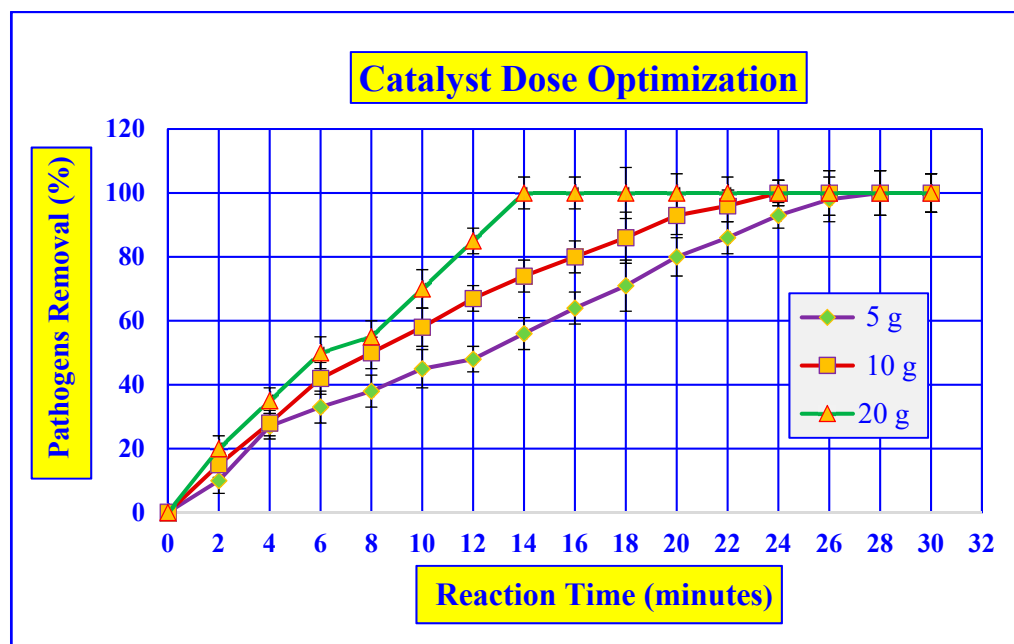


Figure 4. Catalyst dose optimization in catalytic ozonation times ($O_3 = 0.9$ mg/min.; catalyst dose = 5, 10, and 20 g and $T = 30$ min).

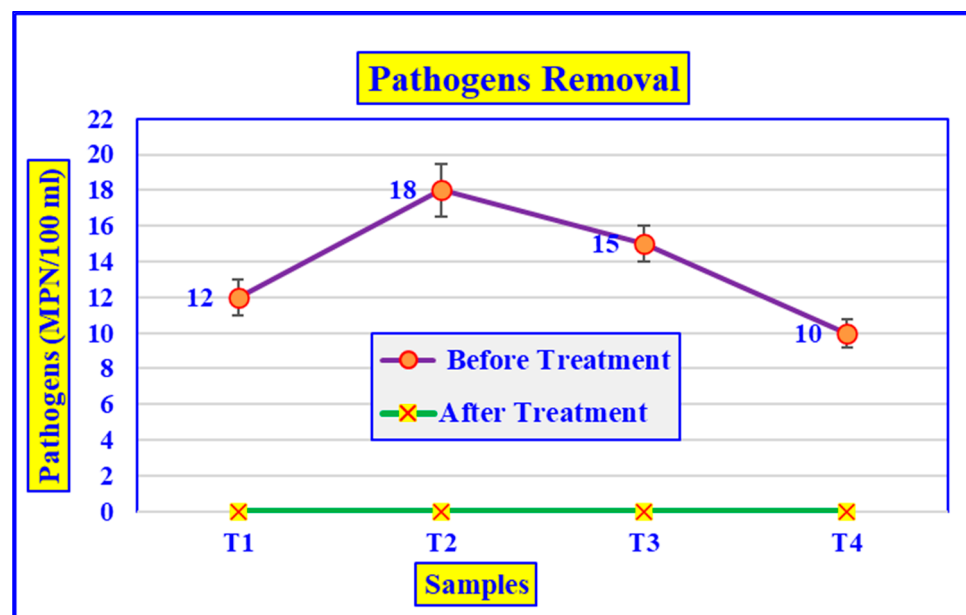


Figure 5. Pathogen removal by catalytic ozonation (initial ozone flow, $O_3 = 0.9$ mg/min; off-gas ozone flow = 0.1 mg/min; O_3 consumption = 0.8 mg/min; catalyst dose = 10 g and $T = 30$ min).

3.5. Arsenic Analysis

Figure 6 shows that arsenic values of all samples decreased by a percentage of 38% to 45%. These results depicted that a combination of catalytic ozonation, activated carbon, rice, and gravel was very effective in the treatment of water with arsenic impurities.

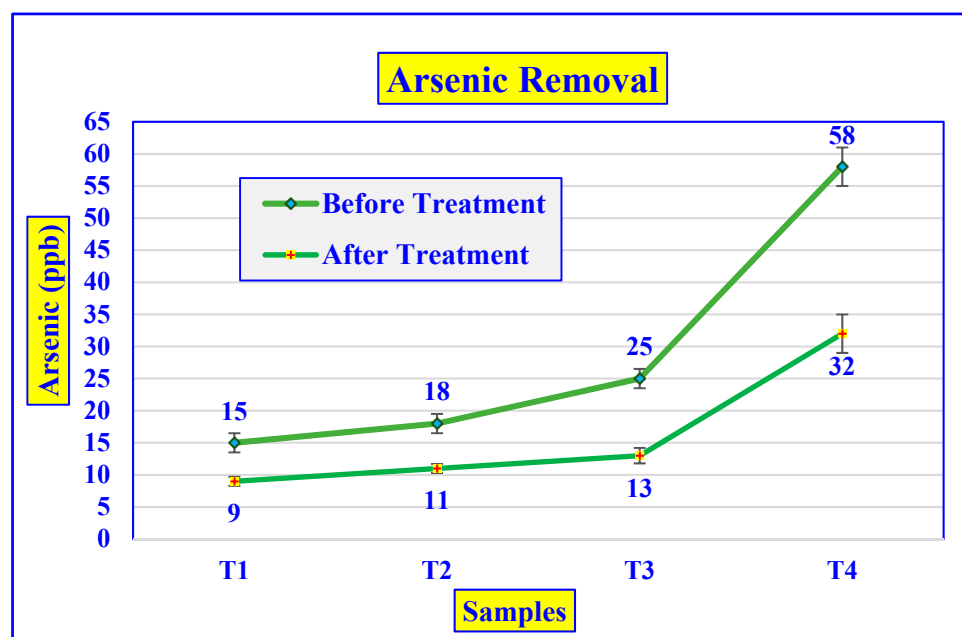


Figure 6. Arsenic removal by hybrid unit (initial ozone flow, $O_3 = 0.9$ mg/min; off-gas ozone flow = 0.1 mg/min; O_3 consumption = 0.8 mg/min, catalyst dose = 10 g; activated carbon = 30 g; rice husk = 60 g; $T = 30$ min).

The chemistry behind this removal is that firstly arsenic is directly adsorbed on the surface of the zeolite. Secondly, as ozone is an oxidizing agent, it precipitates the arsenic after oxidization, and these precipitates are either adsorbed on the surface of zeolite or move to filters used in the second stage.

3.6. Turbidity Analysis

Turbidity is mainly due to the large numbers of individual particles that caused haziness. Turbid water is always very hard to drink. Samples collected from the field were analyzed for turbidity. There was a significant decrease in turbidity of the water-field-collected samples. Figure 7 shows the results, wherein maximum of 90% turbidity removal was achieved. The results confirmed that a hybrid unit was very effective in the removal of turbidity, which is the basic problem in most cases.

3.7. TDS Analysis

For analysis of TDS, samples were analyzed in a laboratory using a TDS meter. The results (Figure 8) revealed a decrease in TDS, and maximum removal efficiency of 27% was achieved. This was primarily due to the adsorption properties of zeolites that salts were adsorbed and the TDS value decreased. To counter-check the obtained results and achieve removal efficiency, samples prepared in a laboratory of known concentration were also analyzed. The results of these samples also showed similar results to those obtained from field samples.

The reason for this partial removal of TDS is ion exchange. Zeolites also exhibit ion-exchange qualities. Due to this ion-exchange property of zeolites, calcium is replaced by sodium. In this way, the quantity of salts is decreased, which results in a reduction of TDS.

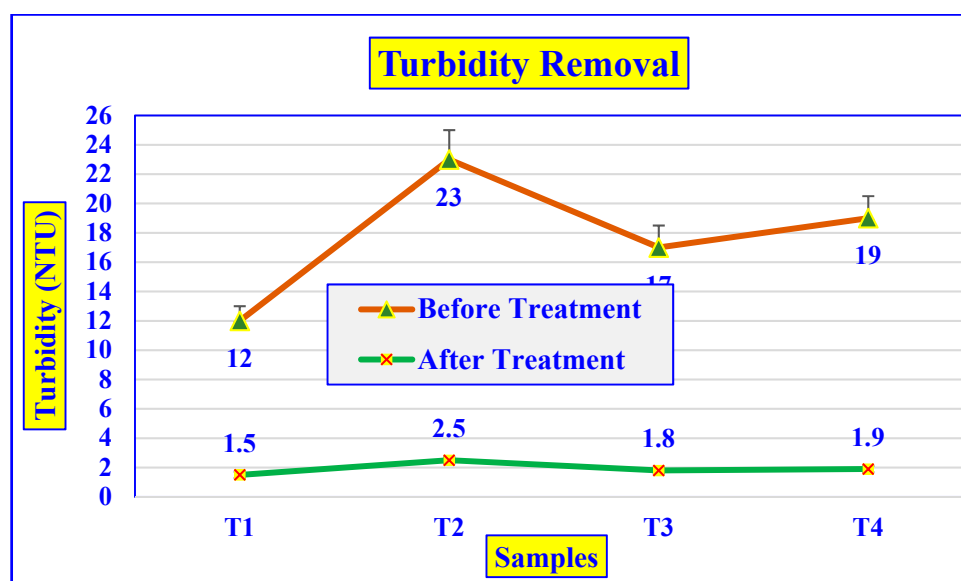


Figure 7. Turbidity removal by hybrid unit (initial ozone flow, O_3 = 0.9 mg/min; off-gas ozone flow = 0.3 mg/min; O_3 consumption = 0.6 mg/min; catalyst dose = 10 g; activated carbon = 30 g; rice husk = 60 g and T = 30 min). Laboratory prepared.

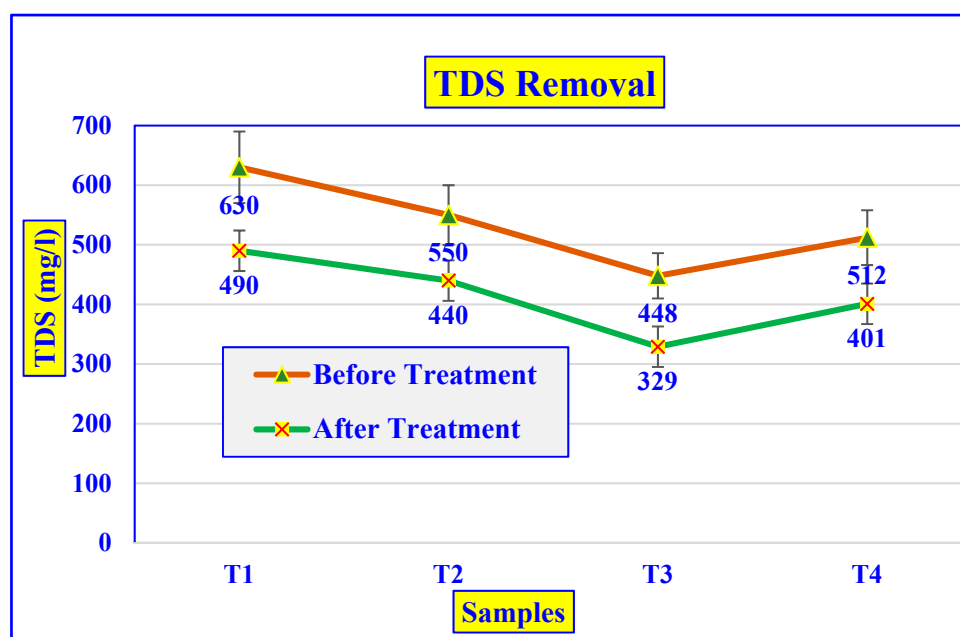


Figure 8. TDS removal by hybrid unit (initial ozone flow, O_3 = 0.9 mg/min; off-gas ozone flow = 0.25 mg/min; O_3 consumption = 0.65 mg/min; catalyst dose = 10 g; activated carbon = 30 g; rice husk = 60 g and T = 30 min).

3.8. Analysis of Challenging Water

The above results revealed that the filter hybrid unit was very efficient in the removal of pathogens, arsenic, TDS, and turbidity individually. To check the efficiency of the designed instrument against a sample that has all these contaminants, challenging water was prepared. The samples of known concentration of all contaminants as described earlier were prepared in the laboratory. Thus, challenging water was a sample that has TDS, fecal coliform, turbidity, and paracetamol. Moreover, a laboratory-prepared sample of 0.05 mg/L concentration of paracetamol was analyzed in challenging water. An internal standard procedure was used to detect recognized pharmaceuticals in the sample. The percentage

removal efficiencies of pharmaceuticals were estimated using the following formula based on the peak area in the sample and the peak area after treatment:

$$\text{Pharmaceutical Removal (\%)} = (A_o - A_t) / A_o$$

where

A_o = initial concentration;

A_t = final concentration.

This challenging water was treated by the same procedure, and the results are shown in Figure 9.

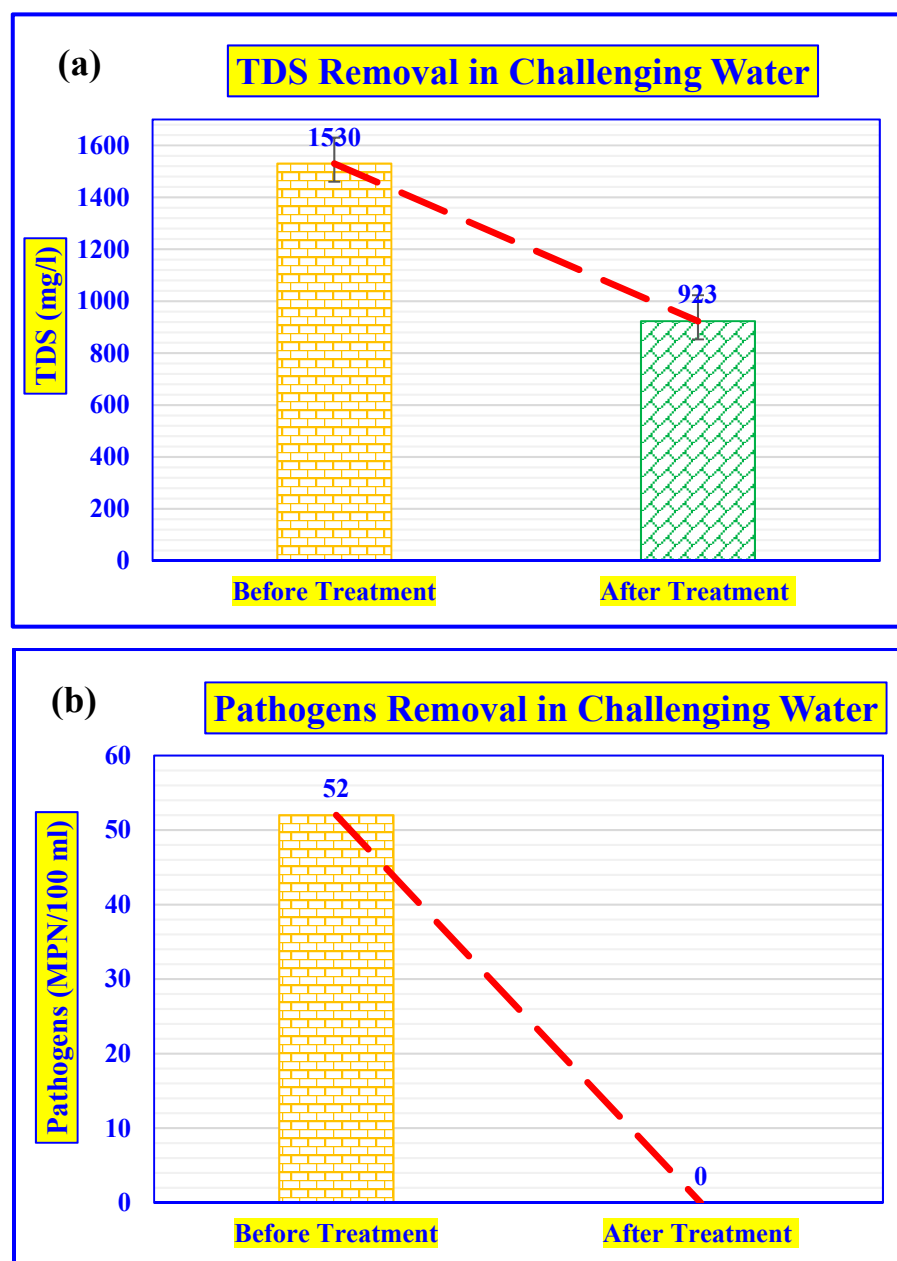


Figure 9. Cont.

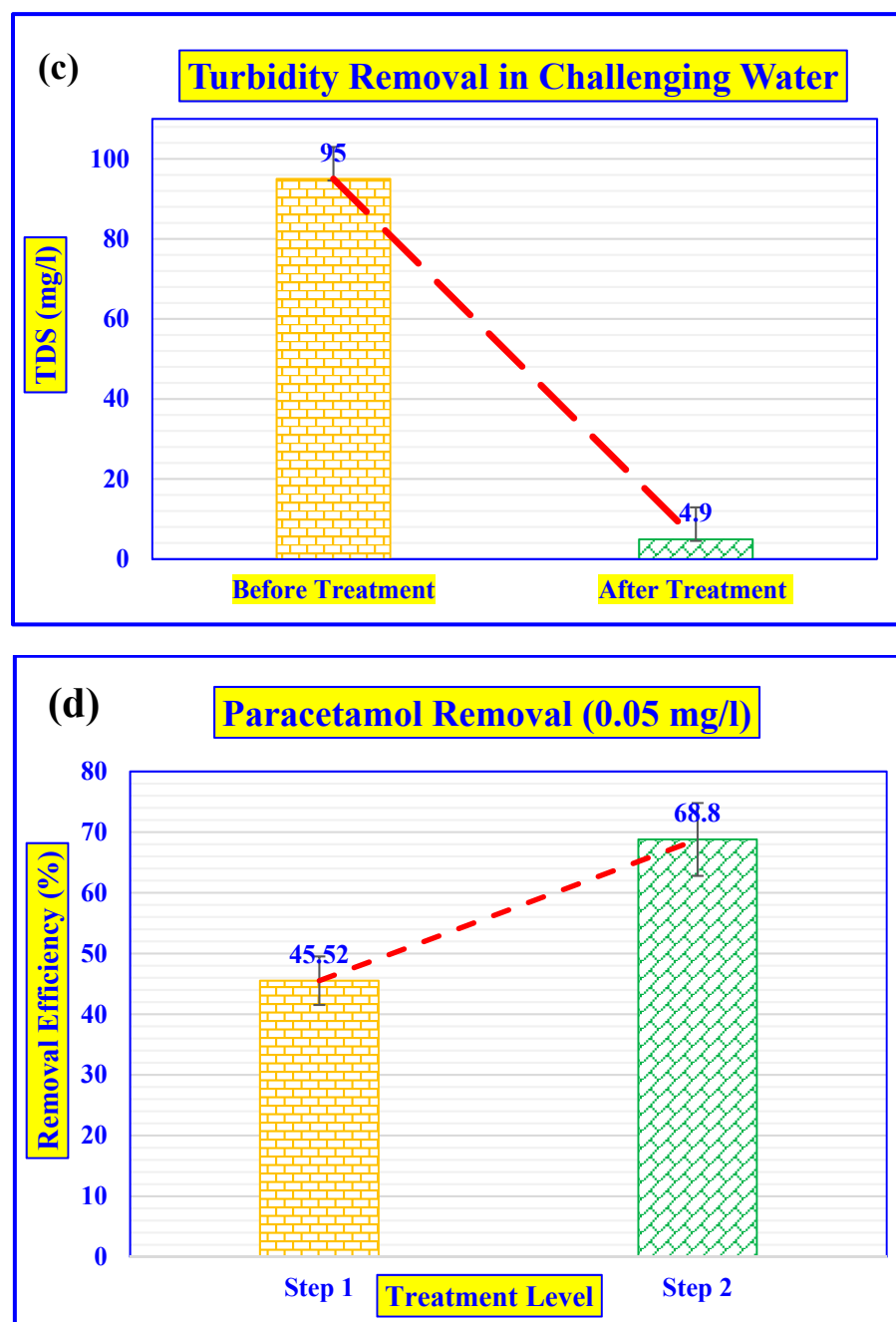


Figure 9. Analysis of lab-prepared challenging water: (a) TDS, (b) pathogens, (c) turbidity, (d) paracetamol (initial ozone flow, $O_3 = 0.9$ mg/min; off-gas ozone flow = 0.02 mg/min; O_3 consumption = 0.88 mg/min, catalyst dose = 10 g; activated carbon = 30 g; rice husk = 60 g and $T = 30$ min).

It has been concluded that the lab prepared challenging water effectively treated by a hybrid unit. The significant removal of TDS, pathogens, and turbidity were 40% (Figure 9a), 100% (Figure 9b), and 95% (Figure 9c), respectively. In addition, Figure 9d depicts the fact that paracetamol was significantly removed (45.5%) by catalytic ozonation. Furthermore, the removal of paracetamol was found at 70% after both stages of treatment. In catalytic ozonation, there are two types of reactions. The first is that molecular ozone is adsorbed on the surface of the catalyst, as well as a pharmaceutical pollutant. Since ozone is an oxidizing factor, the pollutant deteriorated. Secondly, hydroxyl radical as explained earlier also results in the degradation of pollutants.

4. Discussion

In the past, various efforts have been made to treat the drinking water to potable drinking standards. For that, various conventions are applied, such as coagulation, flocculation, adsorption, filtration, and disinfection and advanced treatment methods such as membrane filtration and reverse osmosis to overcome the challenges associated with drinking water treatment. However, these methods have advantages and certain limitations [12,28–34].

Huiqin Zhang et al. (2020) [29] studied the treatment of drinking water by implying one-step ferrate (VI) treatment; in this investigation, turbidity, dissolved organic carbon, and coliform bacteria were studied as target pollutants in drinking water. The initial turbidity from 11.44 NTU was reduced to less than 2 NTU. In the current investigation, the turbidity was reduced from 23 ± 10 NTU to less than 2 NTU in all tap water samples. Moreover, in the current investigation, higher turbidity containing synthetic challenging water was prepared, and its turbidity was reduced from 95 NTU to less than 5 NTU. This suggested that a combination of zeolite-4A-based catalytic ozonation followed by filtration implying rice husk and activated carbon may be a highly efficient process for drinking water treatment in terms of turbidity removal.

Miren Balnco et al. (2019) [35], Huiqin Zhang et al. (2020) [29], and Ankita Dhillon et al. (2018) [33] studied the removal of pathogens in water by implying TiO_2 -doped nanofibrous membrane, one-step ferrate (VI) treatment, and bifunctional nanocomposite, respectively. In these investigations, more than 50% removal of pathogens was obtained. In comparison to the mentioned studies, in the current investigation, fecal coliforms were successfully removed (MPN/100 mL = NIL; Figure 5) from all drinking water samples. The catalytic ozonation followed by filtration removed the pathogen rapidly as compared with the above-mentioned technologies. Moreover, the current hybrid process targets multiple pollutants (TDS, turbidity, fecal coliform, pharmaceuticals, and arsenic) in a single unit. In addition to the above, catalytic ozonation may not produce disinfection by-products, unlike conventional treatment methods such as chlorination [12].

The arsenic was removed from drinking water by various conventional and advanced treatment technologies such as adsorption, filtration, and membranes filtration techniques. Recently, L.E. Verduzco et al. (2019) [28] implied an electrodeposition technique using graphene composites; the results indicate that about 70% of arsenic was removed from the water sample. Moreover, nanofiltration [31], polyurethane foam nanocomposites [36], ion exchange, precipitation, adsorption, and pre-oxidation were successfully implied [30]. The current investigation was in agreement with the previous findings where arsenic was successfully removed (less than the national drinking water quality standards and WHO guidelines; 50 ppb and 10 ppb, respectively) from water by suggesting catalytic ozonation leading to the precipitation of As and followed by removal of As via filtration through rice husk and activated carbons.

Pharmaceuticals are among the emerging contaminants that have been found frequently in drinking waters [37]—they may pose a serious threat to human health and may help in the development of antibacterial microbes. Therefore, it is indeed important to remove pharmaceuticals in drinking waters. The drinking water treatment processes mentioned above [12,28–34] studied various pathogens; however, these studies did not include pharmaceutical removal in drinking water, and therefore there is a need to develop technologies that may remove multiple pollutants in a single unit. In the current investigation, about 70% ($C_0 = 0.05$ mg/L) of paracetamol was removed from a water sample in 30 min (Figure 7). Vincenzo et al. (2018) [37] studied the removal of paracetamol in drinking water by suggesting a photocatalytic oxidation process using ZnO-supported polystyrene pellets. It was observed that about 80% of paracetamol was removed in 240 min. Levgen et al. (2020) [38] studied the removal of pharmaceuticals including paracetamol by using magnetic sol–gel-encapsulated horseradish peroxidase and lignin peroxidase as catalyst. The results found that about 60% paracetamol was removed from water at pH = 3. Therefore, the current investigation showed good performance as compared with some recent findings [39–44].

5. Conclusions and Future Perspectives

Catalytic ozonation provides more effective results as compared to simple ozonation. Catalytic ozonation in combination with filtration is very effective in removing bacteria; pharmaceuticals; and, to some extent, total dissolved solids. In this work, it was found that about 100%, 45%, 40%, 70%, and 95% fecal coliforms, arsenic, TDS, paracetamol, and turbidity removal efficiency was achieved, respectively. Effective removal is achieved when ozonation in combination with zeolites is carried for 24 min and with a 10 g dose of catalyst. Moreover, it is concluded that the studied parameters followed WHO guidelines and NEQS for drinking water quality. Hence, it is concluded that the studied novel hybrid batch reactor is promising for the treatment of drinking water contaminants. The current study may help to achieve sustainable development goals of united nations (SDGs 3, 6, and 9). The research backs up the idea that using a combination of catalysts and treatment processes improves the removal efficiency of various contaminants. The designed treatment combination can be scaled up in the future for household or commercial use.

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