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Degradation of Mononitrotoluene by Electrochemical Method

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ABSTRACT

Present paper deals with studies on the degradation of Mononitrotoluene (MNT) by electrochemical method. The Electro-Fenton and Electro-Peroxide methods are explored to degrade MNT upto its drain discharge limit of 1 ppm. Effects of some important parameters which ultimately decide the rate of degradation of MNT viz. oxidizer, pH, catalyst and voltage etc. have been critically studied. The detailed studies have been carried out which includes variation in different parameters viz. pH from acidic range to basic range, catalyst concentration from 10 ppm to 50 ppm, Oxidizer concentration from 5 mM to 40 mM and potential across electrodes from 4 V to 24 V for efficient degradation of MNT. It is observed that optimised values of precursors viz. catalyst ($FeSO_4$) concentration of 40 ppm, pH of 3, potential across electrodes of 12V and oxidizer (H_2O_2) concentration of 25 mM; Electro-Fenton reaction has been carried out to degrade 50 ppm MNT solution up to its drain discharge limit of 1 ppm and on other hand under Electro-Peroxide reaction results in degradation of MNT from 50 ppm to 12 ppm. The comparative studies of Electro-Fenton and Electro-Peroxide reactions have been carried out for MNT solution and the treated solution has been characterised by using UV-Visible spectrophotometer and Total Organic Carbon (TOC) analyzer and the obtained data on MNT effluent studies may be applicable to explore the efficient mineralisation of 2-Methyl-1, 3, 5-trinitrobenzene effluent. The observed results have been interpreted and reported in the present study.

Keywords: Mononitrotoluene (MNT); Degradation; Electro-Fenton; Electro-Peroxide and UV-Visible spectrophotometer

1. INTRODUCTION

Pollution poses serious threats to both the environment and the organisms. Now-a-days, non-biodegradable and toxic organic contaminants are detected in available useful resources of water on earth. To maintain the purity of potable water is a challenging problem which is faced all over the world. Many organic pollutants are toxic and proved to be carcinogenic even when present at very low concentrations ~ tens of ppm. Basically 2-Methyl-1,3,5-trinitrobenzene is commercially used as energetic materials and being processed like casting and machining which leads to generate said water effluent which is very toxic and leads to adverse effect on environment and ultimately on human beings. As 2-Methyl-1, 3, 5-trinitrobenzene is not easily accessible due to its sensitivity; Mononitrotoluene (MNT) being readily available in the market has been taken as candidature for 2-Methyl-1, 3, 5-trinitrobenzene effluent since a benzene ring is attached with both methyl as well nitro group molecules at different positions. Moreover, the obtained data on MNT effluent studies may be utilised for efficient mineralisation of 2-Methyl-1, 3, 5-trinitrobenzene effluent. Similar to 2-Methyl-1, 3, 5-trinitrobenzene; MNT is also toxic[1] and likely to cause liver damage and also leads to anemia. The safe discharge limit of MNT as well as 2-Methyl-1, 3, 5-trinitrobenzene is 1 ppm.[2] Literature survey indicates that attempts have been made to treat the mononitrotoluene contaminated effluent by different methods that include chemical treatment, bio-degradation, photo-Fenton process and photo–oxidation process using photo catalytic materials. Conventional physical separation methods and biological processes are not economical to degrade organic contaminants in water. Chemical treatment may generate non-energetic secondary waste which could also be toxic and again needs to be treated.[3] On the other hand the biodegradation method is very slow which puts limitations for its use. Activated carbon treatment is feasible, although large quantity of carbon materials is required and also there is no proven method for safe regeneration of the carbon. Carbon column is used for the removal of toxic chemicals from the waste water[4] and carboxymethyl chistosan intercalated montmorikonite (CMCTS-MMT) clay is also used as adsorbent for the removal of cationic dye from the waste water.[5] Recently a method has been developed for the treatment of the energetic compounds contaminated waste water using zero-valent iron nanoparticles (ZVINs)[6], Photo-Fenton process[7] and Photo-oxidation methods[8] are currently gaining much interest but involve use of UV light which is hazardous to eyes and skin etc. and also consumes high electrical energy. Recent developments in the domain of polluted water treatment by chemical and photochemical methods have led to considerable improvement in oxidative degradation procedures for organic compounds dissolved

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or dispersed in aquatic media. These methods are generally referred as advanced oxidation processes (AOP)[9] which is defined as the oxidation process mediated by hydroxyl (OH) radical intermediates. It is well known that the OH radical is highly reactive for the treatment of effluent. The reaction mechanism for the use of oxidizer in acidic region for the treatment of organic effluent is as below:

$$^{\bullet}OH + H^+ + e^- = H_2O, \quad (E^0 = 2.80 \text{ V vs. NHE}) \qquad (1)$$

NHE: Normal Hydrogen Electrode

One remarkable feature of AOPs in comparison to conventional chemical oxidation processes is that the oxidation reaction takes place under ambient temperature and pressure conditions. Most of the photochemical oxidation systems use oxidants such as hydrogen peroxide or ozone usually in the presence of transition metal ion catalyst[10]. Non-biodegradable and toxic organic effluent is completely degraded by AOPs[11]. In Fenton process OH radicals are generated by electron transfer between hydrogen peroxide (H_2O_2) and Ferrous sulphate but Fenton process has some limitations for large-scale of wastewater treatment, which is related to pH i.e. at pH higher than 3, where iron ion precipitated as hydroxide complex which leads to decrease in the efficiency of the process[12-13]. To improve the efficiency for the degradation of non-biodegradable and toxic effluent; Electro-Fenton (EF) process is adopted to treat them upto their drain discharge limit[14]. The objective of present studies is to investigate the efficiency of Electro-Peroxide and Electro-Fenton process for complete oxidation of MNT using Pt electrodes as electrochemical method. We have optimised the process parameters using electrochemical method (electro-peroxide and electro-fenton) to reduce the treatment time and enhance the degradation efficiency of MNT.

2. MATERIALS AND METHODS

MNT, Ferrous sulphate, Sodium chloride, Hydrogen peroxide (30 %) and other chemicals used were of A.R. grade. The Electro-Fenton and Electro-Peroxide reactions have been conducted in a Glass reactor of 1litre volume capacity. The glass reactor has provision for magnetic stirring to make solution homogeneous and current passing through electrodes to initiate the chemical reaction and details are mentioned in one of our earlier publication[15].

In electrochemical experiments, 500 ml of 50 ppm MNT as an effluent has been taken in a glass reactor and oxidizer (H_2O_2), electrolyte (NaCl), catalyst ($FeSO_4$) were mixed and chemical reaction has been initiated by passing current through platinum electrodes dipped in solution. The optimised precursor chemicals for the treatment of 500 ml of 50 ppm MNT are mentioned in detailed at Results and Discussions part of the present research paper. In both the reactions i.e. Electro-Fenton and Electro-peroxide, 0.10 M NaCl is added as an electrolyte to improve conductivity and the ionic strength; which is optimised by doing a series of experiments and details are mentioned in one of our earlier publication[16]. Electro-Fenton reaction has been conducted in the presence of ferrous sulfate catalyst and with pH adjustment. Electro-peroxide reaction has been conducted in the absence of iron salt and also with pH adjustment. Reaction progress as a function of time has been monitored by determining the concentration of MNT content of the solution periodically at an interval of every 30 minutes. Concentration measurements were made using UV-Visible spectrophotometer instrument (Model no.- UV-Visible 3000$^+$ LABINDIA). MNT has been analysed during treatment in the reaction mixture following its degradation kinetics by UV-Visible spectrophotometer. During the Electro-Fenton and Electro-Peroxide treatment of MNT, Total Organic Carbon (TOC) is measured by TOC analyzer (Model no.-ANATOC II Series). The pH measurements has been made using Spectra lab (Model no.- Accu pH-3) instrument. Adjustment of pH of the MNT solution has been decided on the variations in amount of hydrochloric acid and sodium hydroxide with different molar concentrations.

3. RESULTS AND DISCUSSIONS

3.1 Effect of pH

The pH seems one of the crucial factors for Electro-Peroxide and Electro-Fenton process which affect the rate of degradation of effluent. In order to optimize the pH for efficient degradation of MNT; the effect of pH has been studied for a wide spectrum from acidic range (pH of 2) to basic range (pH of 10); provided other parameters were kept constant i.e. catalyst concentration of 40 ppm, applied potential across electrodes of 12 V and oxidizer concentration of 25 mM. Due to instability of H_2O_2 the efficiency of Electro-Fenton process decreases at pH > 4 and the efficiency of process further decreases due to increase in pH from acidic to basic range; this is due to disintegration of H_2O_2 into O_2 and water[17-20] and also the oxidation potential of OH radical decreases due to formation of precipitate of ferric hydroxides complexes.[21-22] At pH > 9, iron complexes were formed in excess amount which reduces the concentration of dissolved iron and overall reduces the efficiency of the process.[20] In the case pH of solution ≤ 2, the effluent degradation has been reduced because H_2O_2 cannot be decomposed to OH radicals by ferrous sulphate[19]. It has been observed that degradation was the highest at pH of 3 for adopted Electrochemical processes. Our results are in good agreements with the results reported by other researchers[14,21]. The overall effect of pH on the degradation of MNT is as shown in Fig. 1.

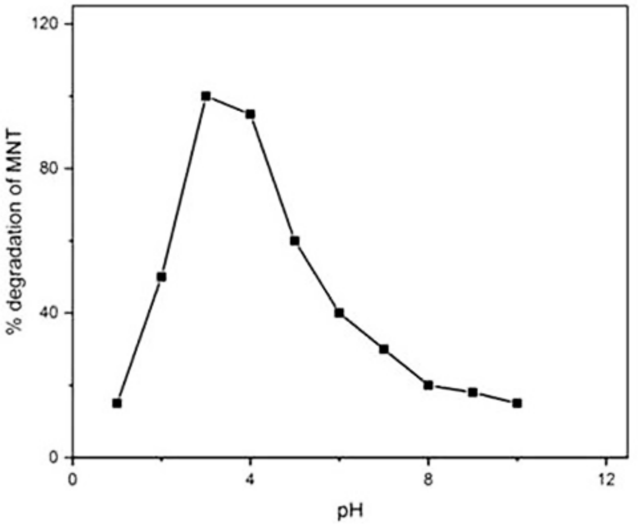

Figure 1. Percentage degradation of MNT as a function of pH.

3.2 Effect of Oxidizer Concentration

H_2O_2 concentration is one of the most important factor in Electro-Fenton and Electro-peroxide reactions which plays crucial role in terms of efficient degradation of MNT. To study the effect of oxidizer on the degradation of MNT; the concentration of oxidizer is varied from 5 mM to 40 mM by keeping other parameters constant i.e. pH of 3, catalyst concentration of 40 ppm and applied potential across electrodes of 12 V. The degradation efficiency of effluent increases with the increase in the hydroxyl radicals concentration as a result of the addition of H_2O_2[22]. For higher concentration of H_2O_2 > 30 mM the degradation efficiency of process decreases, it is due to the combination $OH^{\bullet}$ and $HO_2^{\bullet}$ to form H_2O and O_2[22-24]; which is also shown in equation (2) and (3).

$$OH^{\bullet} + H_2O_2 \rightarrow HO_2^{\bullet} + H_2O \quad (2)$$

$$HO_2^{\bullet} + OH^{\bullet} \rightarrow H_2O + O_2 \quad (3)$$

In the present studies it has been observed that from 20 mM to 30 mM oxidizer concentration is found to be optimal range to degrade MNT from 50 ppm to its drain discharge limit within 2 h. The overall effect of oxidizer concentration on the degradation of MNT is as shown in Fig. 2.

3.3 Effect of Applied Voltage

Applied voltage across electrode is also one of crucial parameters associated with the degradation of MNT effluent for Electro-Peroxide and Electro-Fenton process. To study the effect of applied voltage on the degradation of MNT; applied voltage is varied from 4 to 24 V by keeping all other parameters constant i.e. pH of 3, catalyst concentration of 40 ppm and oxidizer concentration of 25 mM. Voltage is being applied between Platinum electrodes fixed at distance of 40 mm which were kept dipped in solution. It has been observed that below 6 V the degradation of MNT is not completed within specified time and it might be due to insufficient OH radicals formation during chemical reaction. At more than 18 V turbidity was observed, it might be due to complex formation of iron ion hence the degradation efficiency of MNT decreases in the both cases i.e. < 6 V and > 18 V. In the present studies it has been observed that the applied voltage for efficient degradation of MNT was found to be from 6 to 18 V for adopted electrochemical processes. The overall effect of applied voltage on the degradation of MNT is as shown in Fig. 3.

3.4 Effect of catalyst concentration

Catalyst plays very crucial role during electrochemical reaction and hence it is considered as an important factor which affects the Electro-Fenton process. To study the effect of catalyst concentration on the efficient degradation of MNT from 50 to 1 ppm; the catalyst concentration is varied from 10 to 50 ppm by keeping all other parameters constant i.e. pH of 3, applied potential across electrodes of 12 V and oxidizer concentration of 25 mM. It is observed that at 10 ppm catalyst concentration, the degradation of MNT upto drain discharge limit is not completed within 2 h due to insufficient OH radicals formation. For catalyst concentration > 40 ppm, OH radicals are scavenged during chemical reactions as given in equation (4) and iron complex is formed which can be observed in the form of turbid solution[22-23].

$$Fe^{2+} + OH^{\bullet} \rightarrow Fe^{3+} + OH^- \quad (4)$$

Hence the inference is that the optimal range of catalyst is between 30 and 40 ppm for efficient degradation of MNT from 50 to 1 ppm within 2 h. The overall effect of catalyst concentration on Electro-Fenton reaction is as shown in Fig. 4.

3.5 Probable Reaction Mechanism during MNT degradation under Electro-Fenton reaction

The $FeSO_4$ catalyst reacts with hydrogen peroxide (oxidizer) in presence of applied potential and forms OH radicals which further attacks on MNT and intermediate products are formed, these intermediate products further react with OH radicals and converts into eco-friendly by-products (CO_2, H_2O and free N_2 gas) and the overall probable chemical reaction mechanism is as summarised below:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^{\bullet} \quad (5)$$

$$Fe^{3+} + e^- \rightarrow Fe^{2+} \quad (6)$$

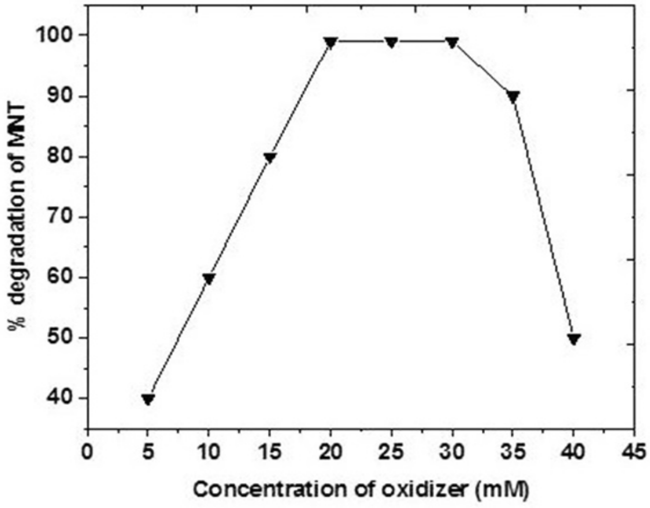

Figure 2. Percentage degradation of MNT as a function of oxidizer concentration.

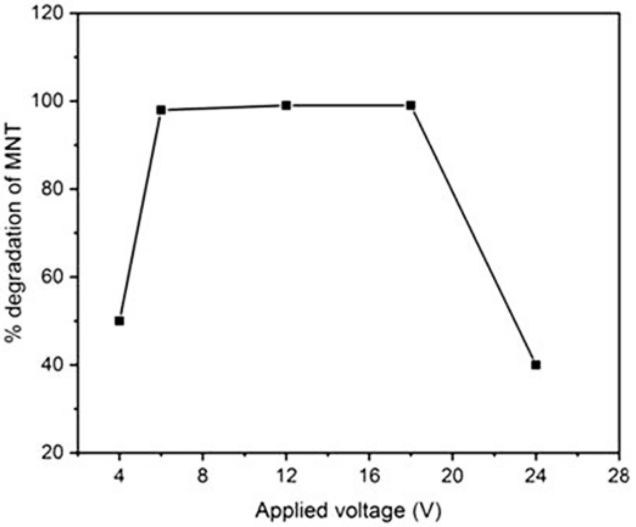

Figure 3. Percentage degradation of MNT as a function of applied voltage.

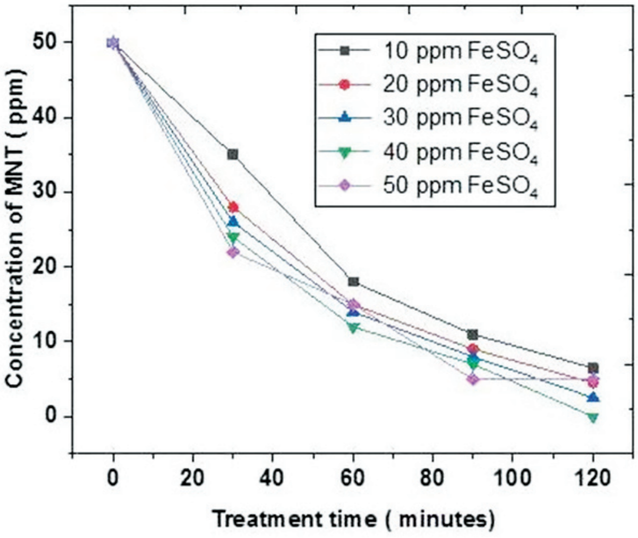

Figure 4. MNT concentration as a function of treatment time using different catalyst concentration.

$\text{MNT} + \text{OH}^\bullet \rightarrow$ Intermediate products (7)

Intermediate products + $\text{OH}^\bullet \rightarrow CO_2 + H_2O + N_2$ (g) (8)

The detailed studies of intermediate products itself is separate topic and is open for discussions. However, 'Recent advances in the electrochemical oxidation water treatment: Spotlight on by product control' is discussed in details by Yang[25].

3.6 Comparative study of Electro-peroxide and Electro-Fenton reaction

A series of Electro-peroxide and Electro-Fenton experiments have been carried out for efficient degradation of 50 ppm of MNT at optimised process parameters viz. pH of 3, oxidizer (H_2O_2) of 25 mM, catalyst ($FeSO_4$) of 40 ppm and applied voltage across electrodes of 12 V. The comparative studies of MNT degradation for Electro-Peroxide and Electro-Fenton processes which yields in diminution of MNT concentration as confirmed by UV-Visible Spectrophotometer and TOC analyser are shown in Fig. 5 and Fig. 6; respectively.

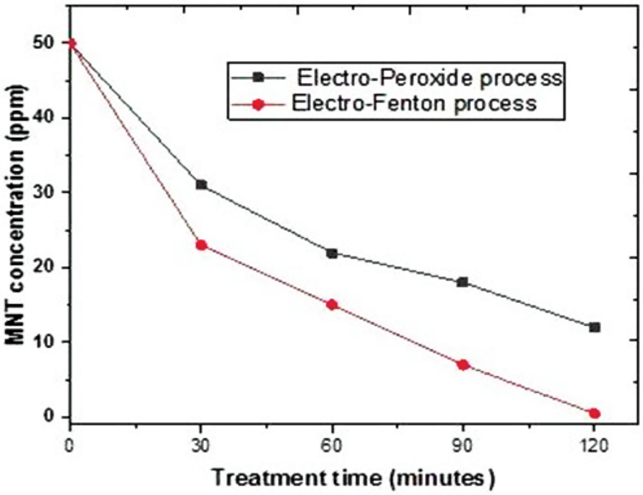

Figure 5. MNT concentration as a function of treatment time for Electro-peroxide and Electro-Fenton Process.

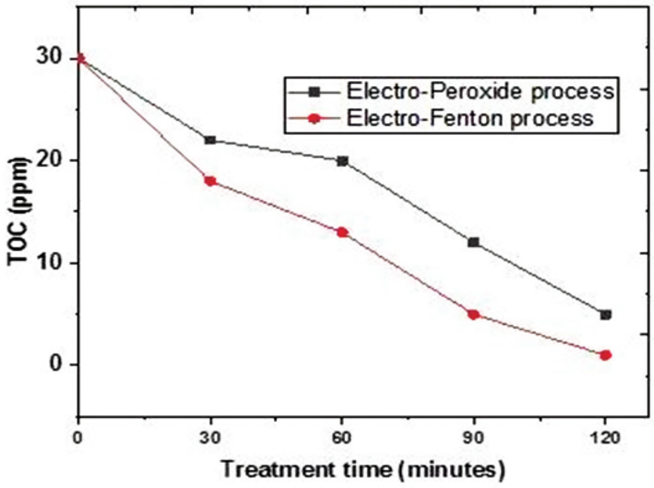

Figure 6. TOC as a function of treatment time for Electro-peroxide and Electro-Fenton Process.

Inference can be drawn from Fig. 5 and Fig. 6 that the Electro-Fenton reaction seems comparatively much faster than Electro-peroxide reaction. During the experiment of MNT treatment by Electro-peroxide method, the solution observed to be turbid due to formation of some intermediates during oxidative degradation which react slowly in the absence of iron salt. This could be a reason for the observed slower kinetics under Electro-Peroxide treatment of MNT. Under Electro-Fenton conditions, these intermediates form complexes with ferric salt and regenerate ferrous ion which results in higher concentration of OH radicals through fast Electro-Fenton reaction as mentioned in equation (5) and (6). As a result of this, MNT concentration decreases at a faster rate under Electro-Fenton reaction. Absence of turbidity in the Electro-Fenton reaction may be due to non-accumulation of intermediates products. This is reflected in the comparable kinetic behavior of Electro-Peroxide and Electro-Fenton reaction as shown in Fig. 5 and Fig. 6. In the present studies it has been found that under Electro-Fenton reaction 50 ppm MNT solution degraded up to its drain discharge limit of 1 ppm within 2 h and on the other hand under Electro-Peroxide reaction, MNT concentration degraded from 50 ppm to 12 ppm within 2 h as shown in Fig. 5. It is also observed that TOC value found decreased from 30 ppm to drain discharge limit of 1 ppm by using Electro-Fenton reaction whereas in the case of Electro-peroxide reaction the TOC is degraded from 30 ppm to 5 ppm as shown in Fig. 6. Overall it has been observed that Electro-Fenton process is superior over Electro-Peroxide process in terms of fast treatment time of MNT within 2 h from 50 ppm to safe discharge limit of 1 ppm.

4. CONCLUSIONS

For MNT effluent degradation; the Electro-Fenton reaction found to be superior in comparison with Electro-peroxide reaction due to rapid and effective degradation of MNT concentration from 50 ppm to 1 ppm of safe discharge limit within 2 h. We have demonstrated a method for MNT effluent treatment at drain discharge limit of 1 ppm by optimising the process parameters viz. pH of 3, catalyst ($FeSO_4$) concentration of 40 ppm, potential across electrodes of 12 V and oxidizer (H_2O_2) concentration of 25 mM, for electro-Fenton which seems more effective than electro-peroxide process. The electro-Fenton process adopted for MNT treatment is simple, rapid and seems cost effective. The data of optimised process parameter for degradation of MNT will be highly useful for 2-Methyl-1, 3, 5-trinitrobenzene effluent treatment which seems a hot topic in an energetic materials industries.

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CONTRIBUTORS

Mr Ratanesh Kumar has completed his MSc in Chemistry from MKU. Since 2005, he is working in the Applied Physics Division, BARC. He is involved in the development of electrolytic technique for the treatment of organic pollutants in water.
In the current study, he carried out electrochemical treatment experiments and optimised the process parameters to treat MNT upto its drain discharge limit within specified time. He analysed the treated samples by various techniques viz. UV-Visible Spectrophotometer and TOC etc.

Dr P.B. Wagh obtained his PhD (Physics) in 1999 from Shivaji University, Kolhapur. He joined BARC in 2002. He is currently working as Scientific Officer 'G' in Applied Physics Division, BARC. His main areas of research include 'Aerogel materials' and 'Energetic material'. He has to his credit about 100 research publications cited in various international and national journals. Presently, he is involved in R&D activities associated with synthesis and characterisation of nano-size energetic materials by Sol - Gel method.
In the current study he proposed idea of electrochemical treatment of MNT, participated in experiments and characterisation of treated samples.

Dr Sanjay V. Ingale has completed his MSc in Physics with specialisation in Materials Science from Shivaji University, Kolhapur. Since 2002, he is working in the Applied Physics Division, BARC. He is involved in development and processing of nano structured energetic materials.
In the current study, he participated in characterisation of treated samples by various techniques viz. UV-Visible Spectrophotometer and TOC etc.

Dr K.D. Joshi received PhD from the University of Bombay, Mumbai, in 2009. He joined BARC Mumbai as a scientific officer in 1993. His current research interests include understanding of the material response under high dynamic and static pressures employing theoretical and experimental tools. The removal/dilution of organic pollutants from water and wastewater is also his current research interest.
In the current study, he availed the process facility and characterisation facilities like UV-Visible spectrophotometer and TOC etc. Guided other authors to conduct the treatment work of MNT by various methods and characterisation of treated samples.