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Effects of faujasite X and Y zeolites on the 1,1,1-trichloro-2,2' bis(*p*-chlorophenyl)ethane (DDT) degradation during water purification

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Abstract: The ability of zeolites to abstract and denature organochloride pesticides finds application in water purification practices. In this study, activated faujasite X and Y zeolites were separately exposed to 1, 2 and 4 ppm concentrations of 1,1,1-trichloro-2,2' bis(*p*-chlorophenyl) ethane (dichloro dipheny trichloroethane - DDT) water solutions. For the 1 ppm DDT solutions, the resultant degradation products and residual DDT were minimal with concentrations in zeolite treatments reducing to below detection limit (0.005 ppm) in about 2 hours. In addition, the rate of dissipation was found to somewhat depend on the levels of DDT concentration and the type of zeolite used. The main degradation product in samples exposed to faujasite X was dichloro dipheny dichloroethylene (DDE) whereas in the faujasite Y exposed samples, both the DDE and dichloro diphenyl dichloroethane (DDD) were obtained.

Keywords: Faujasites, DDT, Degradation Products, Rates, Water Purification

1. Introduction

The primary purpose of any water treatment methods is to make the water fit for the intended purpose. Organochlorinated contaminants from various sources are ubiquitous in the environment [1] and they eventually end up in the water systems [2, 3]. Studies on world river water systems indicate that organochloride pesticides such as 1,1,1-trichloro-2,2' bis(p-chlorophenyl)ethane (dichloro dipheny trichloroethane - DDT) which was legislatively banned about two decades ago is still present in the waters [2,4]. Literature reports that the DDT degrades in the tropical fresh waters to produce 1,1-bis-(4-chlorophenyl)-2,2-dichloroethene (dichloro dipheny dichloroethylene - DDE), 1-chloro-4-[2,2-dichloro-1-(4-chlorophenyl)ethyl]benzene (dichloro diphenyl dichloroethane - DDD), 1-chloro-2,2-bis-(4-chlorophenyl)ethane (DDMS), 1-chloro-2,2-bis-(4-chlorophenyl) ethane (DDMU), and others [5]. It is worth noting that some of the degradation products, DDD for example, are reported to be environmentally more potent toxicants than the parent DDT [6,7]. The persistence and fate of organochloride pesticides such as DDT therefore continues to be of great concern during water purification because any level of concentrations of their residues are known to have potential toxic effects both on human and aquatic organisms [8].

Recently, a number of germane attempts including biological treatments, photochemical reaction, metal ion-catalyzed reactions, zero-valent metals of Fe, Zn, and Ni/Fe systems have been reported [3, 9, 10, 11]. Water purification methods that target organochlorides such as the use of activated carbons are singly ineffectual [12] hence necessitating the modern in-line coupling with zeolites [1]. Essentially, any efficacious decontamination method ought not to produce a more noxious progeny than the parent pollutant. Meaning that during DDT decontamination from wastewaters, a safe technique would be one that only results in less environmentally potent products than the original pollutant in terms of threshold levels and toxicity. More specifically, a complete adsorption of the pollutant or total hydrodechlorination of it; a process that removes all the chlorides from the hydrocarbon molecule, would be the

desired efficacious one [1]. In addition, literature is replete with applications of more siliceous, larger pore zeolites such as the HMS, MCM-4, SBA-15, and MCM-48 [13], and clays and organoclays [7] for elimination of the DDT from wastewaters.

Since the use of adsorption method as a decontamination measure is fraught with possibility of reversal on some chemical reactions should any of the reaction conditions shift, hydrodechlorination remains the most trusted means to decontaminate any organochloride polluted wastewater [1, 14]. Reports are that halogeno-olefins get reaped of their halogens, a process of hydrodehalogenation when they are exposed to a low Si/Al ratio zeolite such as faujasite X (sometimes labeled NaX or X) than to a higher Si/Al ratio one such as faujasite Y (sometimes labeled NaY or Y) [14]. Since the DDT molecule has similar functional groups to those of alkylhalides which were initially used to investigate nucleophilic chemistry in the NaX and NaY [14], this work sought to extend the investigation on the possible roles, in terms of efficiency and removal, of both NaX and NaY in DDT degradation. Here, water from lake Victoria was used as the water source since the communities around the lake do use the water for domestic purposes. Given that the nature of DDT degradation products is, to an extent, determined by prevailing conditions such as the nature of the zeolite used [13], we intended to have the Si/Al ratio and by extension the basicity and acidity of the medium to be the variable factors in this decontamination study. The intension was to eventually use the faujasite X with more Brosnted acidic and Y with less Bronsted acidity [15] in wastewater purification procedures where only adsorption and hydrodechlorination actions occur.

Although, the natural DDT environmental concentrations ever reported are in the range of 0 – 3.5 ppm [2], this work considered concentrations of 1, 2 and 4 ppm DDT exposure levels so as to study the entire range. Due to them being used as the environmental quality reference standards because of their inherent toxicity [16], the sum total of the DDT, DDE and DDD concentrations was the main focus of this study.

2. Materials and Methods

2.1. Chemicals and Instruments Used

The standards of p,p'-DDT, p,p'-DDE and p, p'-DDD (+99% assay) and Florisil (magnesium silicate 60-100 mesh) (+99% assay) were supplied from Kobian Company, Nairobi Kenya. The Nitrogen gas used in the Gas Chromatogram (GC) work was supplied by BOC Company, Nairobi Kenya. Analytical grade n-hexane, acetone, iso-octane and anhydrous sodium sulphate were supplied by Zeta Chemicals Company, Nairobi, Kenya. Faujasite –X (Si/Al = 1.5) and Y (Si/Al = 3.00) zeolite ca. 2 µm particle size were obtained from Sigma-Aldrich Chemical Company, St Louis, MO, USA. The Varian chrompack CP-3800 and 3400 ECD detector GC sourced from Palo Alto, CA, USA and GC-MS (Agilent 6890 GC and 5975 MS) acquired from Santa Clara, CA, USA were used for the analysis and characterization of the samples. For FT-IR measurements, Bruker Equinox 55 spectrometer from Madison, WI, USA was used.

2.2. Experimental Procedure

The lake water used was first stored in the dark at room temperature for two weeks before use. The experimental procedure was adopted from the works of Ref. 9. Whereby the glass containers were placed in the open under a garden-house net at location; 0° 0'S; 34° 36'E, and protected from rain flooding by a plastic sheet held ca. 2 meters above the ground to permit natural air currents. During the experiment, the average air temperature ranged from ca. 25 ^{o}C to 30 °C and daily sunshine of ca. 12 h.

The activation of the zeolite was done under vacuum conditions where the required amount of zeolite was put in a glass wool-plugged sample tube and evacuated by raising the temperature 50 ^{o}C every 30 min to 350 ^{o}C. The set-up was then held at that temperature for 4 h under a 10^{-5} torr vacuum. The activated zeolite was allowed to cool to room temperature then stored as such till use. The experimental treatments were; a) 1 litre of water, b) 1 litre (L) of water + 0.3 g activated zeolites (separately X and Y), c) 1 L of water + varied amount of pesticide (separately 1, 2, and 4 ppm) + 0.3 g activated zeolite (X and Y), and d) 1 L of water + pesticide at 1, 2, and 4 ppm without zeolites. The required amounts of the DDT were first dissolved in 3 mLs of triple distilled acetone before dozing into the experimental waters. After stirring with a glass rod, 40 mL of samples (in the set-ups a) to d) above) in triplicates were obtained at 2, 5, 8, 22, 48, 120, 240, and 720 hours of exposure time. Then the concentrations of DDT and characterization of degradation products were determined by GC and GC-MS analysis respectively.

To avoid the thermal decomposition of the analyte during the analysis, the GC instrument was thermally conditioned, glass inserts were cleaned and the column ends were cut to remove any previous accumulations that would initiate the decomposition. The GC was equipped with nickel 63 ECD detector and CP-SIL 8CB-15 m, and 0.25 mm i.d. The column temperature was programmed at 150 ^{o}C for one min changing at 4 ^{o}C/min to 200 ^{o}C (0 min) and 4.5 ^{o}C/min to 300 ^{o}C. Injector temperature was maintained at 250 ^{o}C and at 300 ^{o}C for the detector. The flow pressure of 30 Psi for nitrogen gas was applied. Sample size of 1 µl in split ratio of 1:20 was used during all the samples analysis. The detection limit of 0.005 ppm for the DDT was achieved during the analysis. Confirmatory analysis was done using a 6890N GC coupled to a 5975 MS (Agilent technologies). A 60 m x 0.25 mm x 0.25 µm i.d, DB5-MS non-volatile residue free column (J&W) was used. The helium carrier gas was applied at a flow of 2 mL/min. The sample injection volume was 2 uL with a split splitless mode. The temperature was programmed at 110 ^{o}C for 1.8 min, rising by 10 °C/min to 180 °C, and then by 12°C to a final temperature of 320 ^{o}C. The Mass spectra were acquired in full scan mode from m/z 50 to m/z 550, at 1 scan/s. A solvent delay for data

acquisition of 2.5 min was used to avoid disturbances in MS detection. The inlet degradation of DDT was checked daily and controlled within 15%. While the standard mix procedure was followed to verify whether the compound detected in the GC-ECD was the actual target compound.

To monitor any molecular anchorage of the DDT or of its degradation products to the zeolite matrix by Infrared spectroscopy, the used zeolite sample pellets were fixed in FT-IR Bruker Equinox 55 spectrometer at a nominal resolution of 2 cm^{-1}. The spectrometer was purged with nitrogen gas for 30 minutes before and after pellet insertion, after which the spectrum was recorded over the 4000 – 400 cm^{-1} range. A total of 128 scans were collected for each sample spectrum.

Recovery of pesticides from the water was also done to determine the extraction efficiencies of the methods used. The recovery experiment was performed by spiking standard p, p'-DDT pesticide into both the lake water and deionised water and each mixture was shaken for about 2 minutes to homogenize. Each spiked mixture was then allowed to settle for ca. 15 minutes after which sample extraction procedure, as explained above, was followed. The extracts were injected into the GC-ECD and used for determination of extraction efficiencies. To obtain the amounts of DDT left adhering to the glass walls at the end of the experiment, the glass walls were three times rinsed with acetone, then the extracts were concentrated in iso-octane and the analysis done using the procedure above.

3. Results and Discussion

3.1. Characteristics of the Water Used

The water used for this study had a background DDT concentration of ca. 0.012 ppm and a pH of ca. 6.9. The pH was continuously monitored during the course of the study and was found to be statistically unchanged in all the treatments. All the data presented below had been corrected for the background DDT concentrations. In the control set-up, no DDT metabolites such as DDE and DDD were found.

3.2. Recovery of the p, p'-DDT from the Sample Waters

Recovery efficiencies of 96.30 ± 0.05 % and 98.31 ± 0.03 % were obtained for *p, p'*-DDT extractable residues in samples of lake water and deionised water respectively. These DDT recovery rates for the Y and X treatments obtained by the n-hexane solvent-solvent extraction method were within reported successful extraction range of 80-120 % [2]. Such high recovery percentages indicate that the extraction methods employed here are acceptable and the values compare closely with the recovery percentages obtained by other workers [2, 5].

In addition, about 0.01 ppm of both the 2 ppm and 4 ppm DDT treatments remained adhered to the walls of sample glass containers. Due to the low amount adhering to the walls, to the best approximations, the effects of the container wall-bound DDT concentration was subsequently ignored. Also, other loses such as through evaporation to surrounding air, photodecomposition, volatilization [17], and adherence to any water-bound impurities were taken care of by the control set-ups. As such, the remaining phenomena possibly impacting on the concentration of the *p, p'*- DDT in the sample solutions were the entrapment within the zeolite cages [14] and the zeolitic catalytic degradation [13].

3.3. Degradation of p,p'-DDT in Water under Different Treatment Conditions

3.3.1. At 1 ppm DDT Concentration

Generally, enhanced dissipation rates were observed for treatments with zeolites. The drop in DDT concentrations was sharper in the first 2 h of reaction and the DDT residual amount went down to non-detectable (n.d) level after ca. 5 h of reaction time (Table 1).

Table 1. *Degradation of 1, 2 and 4 ppm p, p'-DDT in water under different treatments with exposure duration in hours ((-) = Non-detectable).*

Time / hour	Water + DDT / ppm (≈ ± 0.001)			Water + DDT + X /ppm (≈ ± 0.002)			Water + DDT +Y /ppm (≈ ± 0.001)		
	1 ppm	2 ppm	4 ppm	1 ppm	2 ppm	4 ppm	1 ppm	2 ppm	4 ppm
0	0.98	1.98	3.98	0.98	1.98	3.98	0.98	1.98	3.98
2	0.81	1.90	3.65	0.12	0.89	2.71	0.31	0.70	2.56
5	0.80	1.60	3.20	-	0.36	1.91	-	0.62	2.06
8	0.60	1.20	2.61	-	0.20	1.40	-	0.21	1.51
22	0.51	1.04	2.08	-	0.09	1.09	-	0.16	1.15
48	0.49	0.98	1.95	-	0.01	0.99	-	0.13	1.10
120	0.48	0.97	1.60	-	-	0.98	-	0.11	1.10
240	0.47	0.97	1.41	-	-	0.98	-	0.11	1.09
300	0.47	0.97	1.40	-	-	0.97	-	0.10	1.10

The reduction of the DDT levels at 1 ppm to non-detectable levels in just ca. 2 h is consistent with both X and Y having a dual absorptions and catalytic capacities much higher than 0.5 ppm/ h. The observed high absorption capacity was also exemplified when the 0.012 ppm background DDT concentration for the raw water was not detected any time after the treatments with both the zeolites X and Y.

It is likely, as hitherto been reported, that at 1 ppm DDT level, both zeolites X and Y do irreversibly absorb the pesticide [1]. However, water without any zeolite treatments reduced the DDT concentration to ca. 0.51 ppm in 22 h. Thereafter, a concentration of ca. 0.48 ppm of the *p, p'*-DDT was then maintained at an equilibrium to the end of the study. Such an equilibrium situation was noted after ca. 48 h for the 2 ppm (Fig. 1) and after ca. 120 h for the 4 ppm (Table 1) samples.

Ref. 18, also noted such an equilibrium phenomenon and attributed it to an equilibrium being attained between the DDT in solution, DDT adsorbed in surfaces and the DDT volatilized in the ambient air.

Since the normal maximum background environmental DDT contamination level, unless in an event of a major contamination, rarely exceeds 3.50 ppm [2], the results in Table 1 therefore imply that zeolites X and Y can, at 0.03 g/L (adsorbent/wastewater) loading rate, effectively be used to completely clear DDT off environmental wastewaters. More specifically, Table 1 indicates that at 1 ppm of DDT concentration, faujasite X is almost twice as effective as Y in the removal of the pesticide.

3.3.2. At 2 ppm DDT Concentration

At the 2 ppm DDT exposure level (Table 1), the observed DDT degradation in the water for the initial 10 h had a degradation rate of ca. 0.4 ppm/h; translating to ca. 25 % per hour removal rate. Compared to the higher removal rate observed for 1 ppm, the lower rate for 2 ppm DDT level is consisted with the degradation rate being a factor of the loading level of the pesticide. Such a loading level related phenomenon has similarly been reported before, and is possibly related to the adsorption capacity of the adsorbent [13]. In addition, for the X treatment, the concentration reduced to non-detectable limit in 48 h of application time (Fig. 1). However, from the 48th h onwards, the Y treatment still maintained a concentration of ca. 0.11 ppm of the *p, p'*-DDT.

The result in Fig. 1 shows, further, that the concentrations of *p, p'*-DDT in the presence of both X and Y zeolites decreased with time. From the Fig. 1, out of the initial 2 ppm of the DDT applied into the Y treatment, only 0.70 ppm was water bound at the end of the first 2 h, meaning that some 1.30 ppm of *p,p'*-DDT had either been degraded or just adsorbed into the zeolite. The initial (0-10 h) *p, p'*-DDT decontamination rate was statistically similar for both the X and Y treatments (Fig. 1). However the rate for X treatment later picked up and the concentration of the *p, p'*-DDT was reduced to non-detectable (n.d) level at ca. 50 h exposure time.

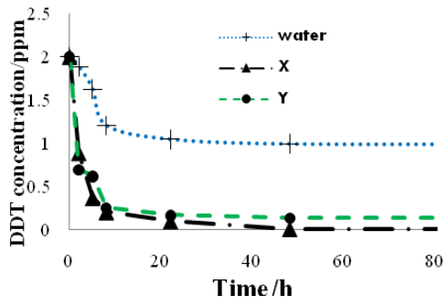

Fig 1. *Amount of 2 ppm p, p'-DDT remaining in solution with time in water and in faujasite Y and Faujasite X treatments.*

Again, Fig. 1 is consistent with X being more effective in removal/degradation of DDT than Y. By convention, the calculated first order half-life of ca. 30.0 hours for the 2 ppm concentration sample is considerably better than half-life of 145 days in field conditions in Nairobi [19], 56 days in fresh lake water [20] and 28 days in river water [21]. However no binding comparison between the above literature values can unambiguously be established because the conditions such as analytical method, sampling procedures and time of pesticide application, under which the studies were done would be of interest.

3.3.3. At 4 ppm DDT Concentration

As was the case for 2 ppm DDT level, at the 4 ppm level enhanced DDT removal rates were similarly observed for the water dosed with the zeolites. For the X and Y treatments, the rates were faster in the first 10 h of reaction time as compared to the untreated water (Table 1).

At the 4 ppm (Table 1), for both water and the X and Y treatments, there were indications of saturation as depicted by an equilibrium situation after ca. 120 h. Both X and Y treatments failed to get rid of all the DDT even after the 300 h of agitation. Specifically, for zeolite X treatment, the DDT concentration reduced to ca. 1.0 after 120 h of application. For the Y treatment, after the initial 48 h, it maintained a concentration of ca. 0.98 ppm of the *p, p'*-DDT as compared to ca. 1.1 ppm of untreated water as shown in Table 1.

3.4. Degradation Products for the 2 ppm DDT Concentration Level

Since the 1 ppm DDT treatment level appeared too little a concentration for the high adsorption and catalytic capacity of the faujasites, while the 4 ppm level seems to overwhelm the capacity of the zeolites, our subsequent considerations in this section focuses only on the 2 ppm level which, in this case, is considered suitable DDT concentration. Many reports detail that the nature of DDT degradation products is determined by, among other factors, the nature of the medium, in this case the nature of zeolites, and the time duration of exposure [1, 9, 13, 22].

3.4.1. Zeolite Y Treatment and DDT Degradation

From the literature, dichloro dipheny dichloroethylene (DDE), dichloro diphenyl dichloroethane (DDD), 1-chloro-2,2-bis-(4-chlorophenyl)ethane (DDMS), 1-chloro-2,2-bis-(4-chlorophenyl) ethane (DDMU) are

some of the DDT degradation products expected in an aqueous environment [2, 5, 9, 22]. However, only DDE and DDD were detected after treatment with Y. During the study, a considerable decrease in the concentration of *p, p'*-DDT remaining in the Y treated samples was also observed (Fig. 2). From the Fig. 2, the *p, p'*-DDE concentration which was initially higher than that of the *p, p'*- DDD, reduced in magnitude and at ca. 18 h, was overtaken by the concentration of *p, p'*-DDD. At the same time, the concentration of *p, p'*-DDD kept on increasing for the duration of the study even though this concentration remained comparatively lower than that of the remnant *p, p'*-DDT (Fig. 2). The observed relative intensities among the products is a phenomenon associated with a series type of reaction mechanism where DDT degrades to DDE which finally produces DDD [23]. It is worthy to note that both the *p, p'*-DDD and the *p, p'*-DDE were simultaneously detected at the beginning of the study with the concentration of *p, p'*-DDD increasing while that of *p, p'*-DDE decreasing (Fig.2). The observed trend is attributable to the hydrodechlorination of DDT to *p, p'*-DDE and eventually to *p, p'*-DDD. The almost instantaneous appearance of the breakdown products at the onset of the study is unique in an aqueous medium. Ref. 9, could only detect the DDE and DDD after 30 days of DDT degradation in a zeolite-free aqueous environment. Since the degradation of DDT as shown in Fig. 2 is consistent with a consecutive/series mechanism [23], the instantaneous appearance of the products implies highly improved rates of reaction for these products in the presence of zeolites. In addition, the relative increase in DDD concentration is indicative of a faster conversion rate of DDE to DDD than the rate for DDT converting to DDE [23].

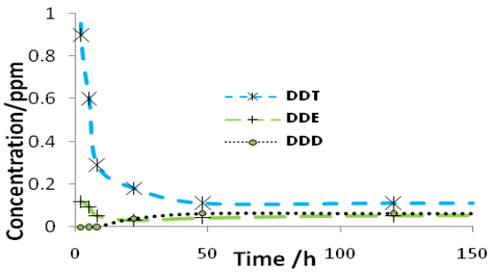

Fig 2. *The concentration of the p, p'-DDT and its degradation product with time under zeolite Y treatment.*

The plateau section (48th hour onwards) in Fig. 2 is a steady state regime where the forward and backward rates balance out [23]. At the end of the experiment (300 h), the major DDT breakdown product was *p, p'*-DDD. Many previous studies have similarly reported the dominance of the DDD over DDE and other breakdown products under aquatic and marine conditions [1, 9, 22]. Since the DDD is reported to be more environmentally potent toxicant than the parent DDT [7], this study indicates that the Y zeolite (which may represent the chemistry of most siliceous zeolites) may not be a good medium for domestic water purification.

3.4.2. Zeolite X treatment and DDT degradation

Unlike in the Y treatments where both the DDE and DDD were detected, Fig. 3 shows that only *p, p'*-DDE was detected in the X treatments where its concentration peaked at 5 h after exposure and then reduced with time to n.d level at ca. 20 h.

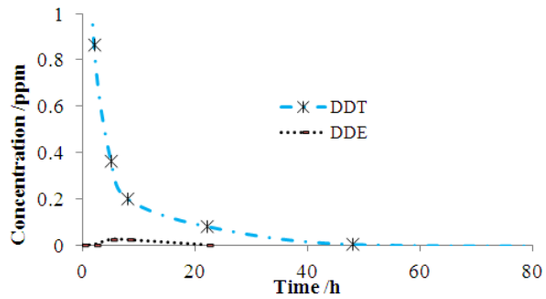

Fig 3. *The concentration of the p, p'-DDT and its degradation products in faujasite X treatment.*

The increase from the 2nd hour and subsequent decline from 5th hour for the concentration of *p, p'*-DDE, is attributable to the generation [14] of the DDE followed by it being adsorbed and eventually decomposed [13] by the zeolite X. However, the ensuing mechanism for the lose in mass balance is not yet apparent. From the data, it can be reasoned that faujasite X favors the formation of *p, p'*-DDE, which was consequently adsorbed within the zeolite matrix. Infrared screening of the characteristic zeolite single four ring (S4R) bands [24] occurring at 761 ± 2 cm^{-1} in NaX shifted to 750 ±2 cm^{-1} when NaX was exposed to the DDT (see Fig. 4), a shift quite consisted with molecular attachments to the zeolite framework [24].

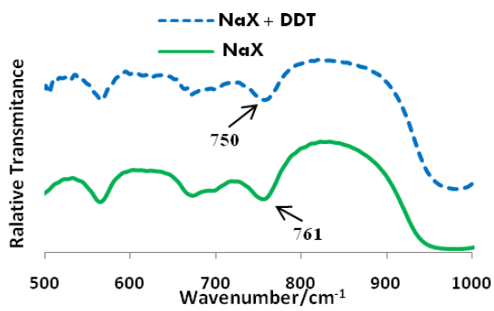

Fig 4. *The IR spectra of the rings vibration region of faujasite X, NaX (dotted line), NaX exposed to 2 ppm DDT (solid line).*

The molecular attachment would imply that apart from catalyzing the degradation of the DDT, zeolite possibly adsorbs both the DDT and its degradation products. This finding is consistent with that of Ref. 14, which observed that degradation products adsorb to the zeolite framework, and cause the S4R characteristic bands to shift. The attachment to the framework possibly impedes further degradation of the DDE to DDD. Notably, as opposed to Y treated samples where the *p, p'*-DDT could be detected for longer durations of the study, in X treatments the decline

was sharper and the concentration reduced to n.d level after 48 h (Fig. 3). Consequently, and for the benefit of domestic water purification, this work asserts that the zeolite X (with more bronsted acidity) is more (devoid of DDD produce) efficacious. Specifically, in both the zeolite Y (Fig. 2) and X (Fig. 3) treatments, the concentration of the common product; *p, p'*-DDE, peaked after 5 h exposure time. The common peaking time for the *p, p'*-DDE is indicative of a common mechanism operating in both Y and X media resulting in the formation of the *p, p'*-DDE.

4. Conclusions

The 1 ppm and the 4 ppm DDT loading levels were found to respectively be inadequate and excessive for both the catalytic and adsorptive capacities of the X and Y zeolites. All the treatments consistently showed faster initial rates for both Y and X treatments. Specifically, the DDT removal rates of ca. 0.5 ppm/ h, 0.4 ppm/h, and 0.2 ppm/h were recorded for the zeolite X treatments on the 1, 2 and 4 ppm levels respectively. At the 1 ppm, both X and Y treatments reduced the concentration of the DDT to below detection limit (0.005 ppm) in 2 h. However, reduction rate for 2 ppm was slower and could only attain below detection-limit concentration in ca. 22 h. The overall zeolite performance showed X being more effective in getting rid of the DDT and its products from the waters. In addition, the X treatments resulted in more environmentally benign degradation products than the use of Y. However, this work could not establish what happens during the first few seconds of the reaction and the presence or absence of possible volatile DDT degradation products which occur in the aquatic environment.

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