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Application of Raw and CTAB Activated Bentonite in the Treatment of Ammonia-Phenolic Wastewater

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ABSTRACT

Adsorption with bentonite offers an efficient, cost-effective and environmentally friendly method for the treatment of ammonia-phenolic wastewater. Therefore, raw bentonite and organoactivated bentonite with hexadecyl trimethyl ammonium bromide (CTAB) were used as adsorbents for the removal of total ammonia, total phenols and total cyanides from untreated ammonia-phenolic wastewater. Better percent removal of total ammonia (34.64%), total phenols (42.50%) from ammonia-phenolic wastewater was achieved with CTAB activated bentonite compared to raw bentonite. Raw bentonite is recommended for the removal of cyanide ions from ammonia-phenolic wastewater over CTAB activated bentonite. Although both adsorbents give a similar percentage of removal, raw bentonite is considered a cheaper option compared to activated due to additional cost and time, so it would be the choice for removing these ions.

Keywords: Raw Bentonite, CTAB Activated Bentonite, Ammonia Nitrogen, Phenols, Cyanides

I. INTRODUCTION

In the last three decades, the pollution of the environment by chemical or biological contaminants has grown rapidly and has become a major concern of today's modern society, governments as well as industry [1]. Wastewater generated as an unwanted product from industry or the domestic includes mainly dissolved and suspended organic solids [2]. Economically, effective wastewater treatment has important effects on saving water, and preventing unnecessary water losses [3]. Bonetta et al. 2022 reported that today only about 0.59% of wastewater in the world as well as 2.4% of wastewater in Europe is reused [4]. Treated wastewater was found to be a more applicable and environmentally friendly option than untreated wastewater [5]. These technologies include adsorption, chemical precipitation, ion-exchange, membrane filtration, coagulation, flocculation, flotation and electrochemical methods [6]. Among various water purification, adsorption is one of the cheapest and fastest methods, where the number of low cost adsorbents in this field is rapidly increasing [7]. The major obstacles of the adsorption methods are the ability to remove different ion types concurrently, high retention time, and cycling stability of adsorbents [8]. Its practical applications in industry and environmental protection are of paramount importance [9]. So modelling of experimental data from adsorption processes is a very important means of predicting the mechanisms of various adsorption systems [10]. Laboratory investigations show that rates of adsorption of persistent organic compounds on granular carbon are quite low [11]. Coke plant wastewater (CPW) is an intractable chemical wastewater and the basic substances found in this waters are: oils and tars, phenols (2000 mg/L), ammonium nitrogen (600 mg/L), total nitrogen (900 mg/L), rhodium (30 mg/L), free cyanides (5 mg/L) and sulphides (50 mg/L) [12], [13]. CPW are immediately directed to purification, and the most commonly used method of purification of such waters is biological, which in most cases is insufficient [14]. The wastewater that is rich in ammonia nitrogen would inhibit the natural nitrification, cause water hypoxia, result in fish poisoning, decrease the water purification capacity, and finally do great harm to the water environment [15]. Ammonia nitrogen (NH4+-N) is the major form of nitrogen present in wastewater, and the control of NH^{4+} -N discharge in wastewater has been a major concern around the world [16]. Ammonia toxicity to aquatic animals occurs when nitrates react with hemoglobin causing a lack of oxygen in their body (methemoglobin) resulting in death [17]. Phenolic compounds reach water streams from various industries such as coal conversion, resin, plastic and petroleum refineries. Therefore, the total amount of phenolic water in the circulating water supply system amounts to 0.2–0.3 m^3 per 1 ton of coke [18]. Under the influence of microorganisms, phenolic compounds, which make up 60-80% of oxygen-poor wastewater, are degraded into CO_2, CH_4 and other compounds [19]. These are toxic organic environmental pollutants that threaten human health, and some of them are suspected of being carcinogenic [20]. The toxic level of phenol for aquatic animals is between 9-25 mg/L, while for humans it is between 10-24 mg/L. It is important to note that the lethal concentration of phenol for animals and humans is between 150 mg/100 mL [21]. The European Union also regards several phenols as priority pollutants and the 80/778/EC directive regulates total phenols in drinking water to <0.0005 mg/L [22]. Ahmaruzzaman and Sharma, 2005 investigated the removal of phenol from wastewater using coal, residual coal, and residual coal treated with H_3PO_4 where the initial concentration of phenol was 1000 ppm [23]. Cyanide is extremely harmful but is common in nature [24]. The typical sources of cyanide contamination are industrial waste from plating and mining industries, burning coal and plastics, and effluent from publicly owned treatment works [25]. Different forms of cyanide include: free cyanide, cyanide ion, cyanide salt, metallocyanide complexes and synthetic organocyanides, also known as nitriles and total cyanide [26]. Cyanide causes rapid breathing, tremors, and other neurological effects and long-term exposure to cyanide cause weight loss, thyroid effects, nerve damage and death [27]. U.S. EPA standards for drinking and aquatic-biota waters regarding total cyanide are 200 and 50 ppb, respectively, where total cyanide refers to free and metal-complexed cyanides [28]. Bentonite is absorbent aluminium phyllosilicate clay [29]. It is an abundant, widely available and low-cost adsorbent for water and wastewater treatment [30]. High specific surface area, chemical and mechanical stabilities, layered structure, high cation exchange capacity (CEC), tendency to hold water in the interlayer sites, and the presence of Brønsted and

Lewis acidity have made clays excellent adsorbent materials [31]. Bentonite is a mineral alumina silicate hydrate included in pilosilikat, or layered silicates. General chemical formula bentonite is $Al_2O_3.4SiO_2.H_2O$ [32]. Hexadecyl trimethyl ammonium bromide (CTAB) can be well compatible with cationic, nonionic and amphoteric surfactants. CTAB has excellent permeability, flexibility, emulsification, antistatic, biodegradable and bactericidal properties. Good chemical stability, heat resistance, light resistance, pressure resistance, strong acid and alkali resistance. It is used as a natural, synthetic rubber, silicone oil and emulsifier, flocculant and softener of synthetic fibers, natural fibers and glass fibers. This product is a cationic surfactant [33]. Yuliana et al. 2020 used CTAB activated bentonite to reduce the use of acids in bentonite activation and to maintain oil quality during refining and storage [34]. Ворончак et al. 2014 found that the activation of bentonite with CTAB enables the reduction of the average particle size due to the partial layering of schiz aluminum silicate [35]. Weng et al. 2021 reported that the time required to reach adsorption equilibrium of bentonite before and after modification with CTAB decreased from 12 h to 2 h, and the adsorption rate increased from 26% to 85% [36].

II. METHODS AND MATERIAL

Ammonia-phenolic wastewater

Ammonia-phenolic wastewater created as a result of the production of ammonium sulfate practically becomes wastewater from which it is impossible to extract any semi-product or product using an economically justified technological process. The untreated (raw) samples used in this experimental study were taken before the treatment of such wastewater. In this experimental study, the concentration of total ammonia, phenols and cyanide in wastewater samples was determined, and the results of that analysis are presented graphically (**Figure 1**., **Figure 2**., **Figure 3**.) in Results and Discussion.

Bentonite

The raw (unactivated) bentonite used in this research work is from the Šipovo mine, Bosnia and Herzegovina. Đozić et al. 2022 in their research announced that the chemical composition of analyzed natural bentonite from Šipovo, Bosnia and Herzegovina by X-ray fluorescence is the following: the highest content of SiO_2 oxide is 48.28 mass % and Al_2O_3 oxide is 23.04 mass %. In addition to these two most abundant oxides, others were also recorded in smaller percentages [37]. Again Đozić et al. 2022 in another experimental study gave the complete chemical composition of bentonite from Šipovo mine expressed through oxides of the corresponding metals [38].

Tabela 1. Chemical composition of Ca-bentonite expressed through oxides of the corresponding metals according to Đozić et al. 2022

Oxide content	mas, %
SiO_2	48,28
Al_2O_3	23,04
TiO_2	0,84
Fe_2O_3	4,52
K_2O	0,29
Na_2O	0,22
P_2O_5	0,014
MnO	0,018
CaO	5,92
MgO	1,98
SO_3	<0,02

Hexadecyltrimethylammonium bromide, CTAB

The CTABCTAB ($C_{19}H_{42}BrN$) used in this work had a purity above 98%. It is a light yellow to white crystalline powder. Whose structural formula is:

Fromula 1. Hexadecyltrimethylammonium bromide, CTAB

Preparation of raw bentonite

The bentonite taken from the Šipovo mine is first washed several times with distilled water, then filtered to remove possible impurities and dried at 70 °C for the next 3 hours. Bentonite dried in this way is sieved through < 75 μm sieve and stored in an escicator for later use and is called raw bentonite.

Preparation of CTAB solution

CTAB solution was prepared by dissolving 2.27 g of crystalline powder in 75 ml of distilled water.

Activation of raw bentonite with CTAB

CTAB solution was added to the suspension in which 5 g of pre-prepared bentonite was dissolved in 100 mL of distilled water. The resulting suspension was stirred on a magnetic stirrer for the next 24 hours. After filtering and washing with distilled water. Br^- was reacted with $AgNO_3$, 0.1 mol/L. The suspension was then dried at 105 °C for the next 1 hour. The activated CTAB bentonite thus obtained was then ground into a fine powder and stored in a desiccator.

Treatment of ammonia-phenolic wastewater with raw and CTAB-activated bentonite

To remove organic components, 3.5 g of raw bentonite was added to 750 mL of ammonia-phenolic wastewater sample. The samples were mixed on a magnetic stirrer for 20 minutes at 1200 rpm. After filtering, the concentrations of total ammonia, phenol and cyanide were determined. In this experimental research, the treatment of ammonia-phenolic wastewater with CTAB activated bentonite was performed by adding 0.5 g of activated bentonite to 100 mL of wastewater at the same process parameters as for raw bentonite.

The percentage of anion removal was calculated according to the following equation:

$$\% = 100(C_i - C_{res})/C_i$$

where is:

C_i - initial concentration of anions in ammonia-phenol wastewater

C_{res} - residual concentration of anions in ammonia-phenol wastewater after treatment with adsorbens

III.RESULTS AND DISCUSSION

Figure 1. shows the results of total ammonia (mg/L) in untreated ammonia-phenolic wastewater.

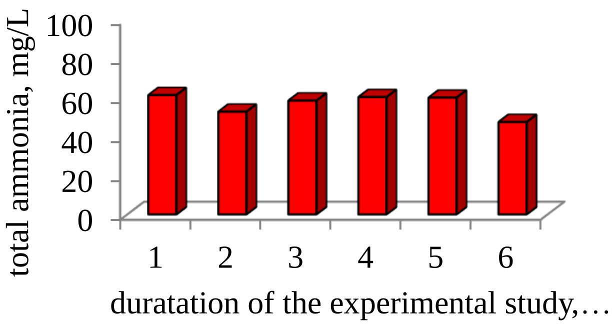

Figure 1. Total ammonia (mg/L) in untreated ammonia-phenolic wastewater during of the experimental study, days

The highest value of total ammonia was recorded on the 1st day of the experimental study and that value was 61.2 mg/L, while the lowest value was recorded on the last day of the experimental study and was 47.4 mg/L. The mean value of total ammonia in the ammonia-phenolic wastewater during the 6 days experimental study was 56.63 mg/L.

Figure 2. shows the results of total phenols (mg/L) in untreated ammonia-phenolic wastewater.

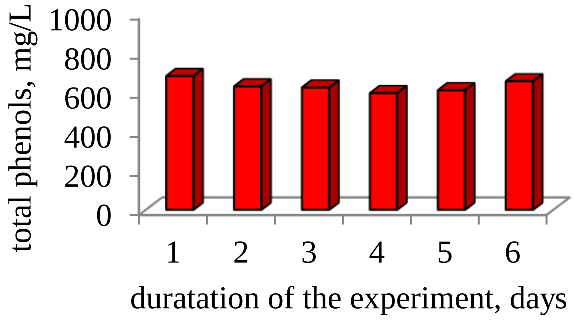

Figure 2. Total phenols (mg/L) in untreated ammonia-phenolic wastewater during of the experimental study, days

The highest value of total phenols was recorded on the 1st day of the experimental study and that value was 683 mg/L, while the lowest value was recorded on the 4th day of the experimental study and was 595,6 mg/L. The mean value of total ammonia in the ammonia-phenolic wastewater during the 6 days experimental study was 633,15 mg/L.

Figure 3. shows the results of total cyanides (mg/L) in untreated ammonia-phenolic wastewater.

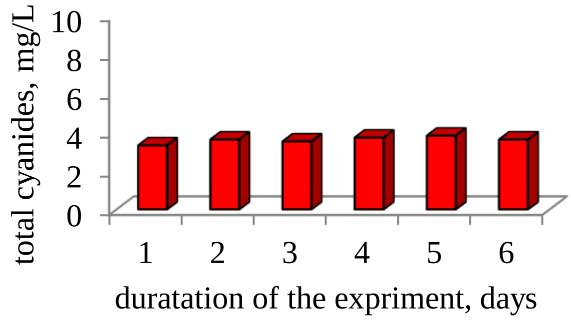

Figure 3. Total cyanides (mg/L) in untreated ammonia-phenolic wastewater during of the experimental study, days

Total cyanides in ammonia phenol wastewater had a mean value of 3.58 mg/L. Against total ammonia and phenol, the lowest value of 3.3 mg/L was recorded for cyanide on the 1st day of the experimental study.

Figure 4. shows the efficiency of total ammonia removal, % depending on the duration of the experimental study, in days.

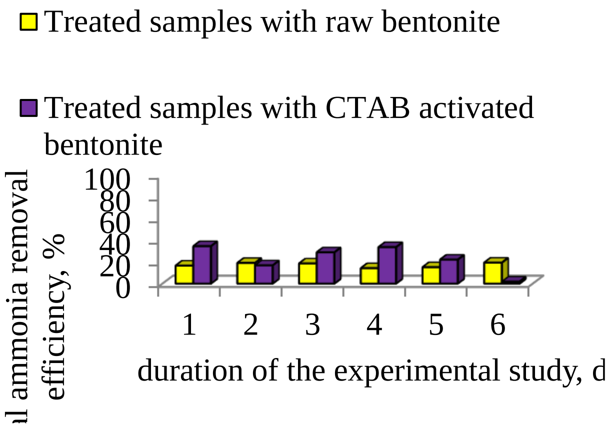

Figure 4. Total ammonia removal efficiency, % depending on the duration of the experimental study, days

Treatment of ammonia-phenolic wastewater with raw bentonite resulted in the sorption of these ions, reducing the residual concentration of total ammonia. Thus, the highest removal efficiency was recorded last day of the experimental study and was 19.40%. Eturki et al. 2012 reported that at pH 6, up to 90% of ammonia was removed with bentonite [39]. Tilaki et al. 2012 reported that as much as 25 to 45% of ammonia can be removed with raw bentonite (depending on sorbent dose and sorbate concentration) [40]. By treating ammonia-phenolic wastewater samples with CTAB-activated bentonite, slightly higher percentages of ammonia ion removal were obtained. The highest percentage of ammonia removal was recorded on the first day of the experimental study and was 34.64%. Based on the experimental results, it is noticeable that a higher percentage of removal was recorded at higher initial concentrations of total ammonia in untreated samples. **Figure 5.** shows the efficiency of total phenols removal, % depending on the duration of the experimental study, in days.

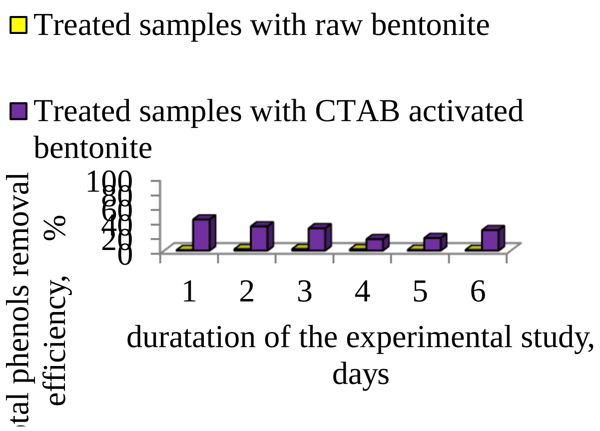

Figure 5. Total phenols removal efficiency, % depending on the duration of the experimental study, days

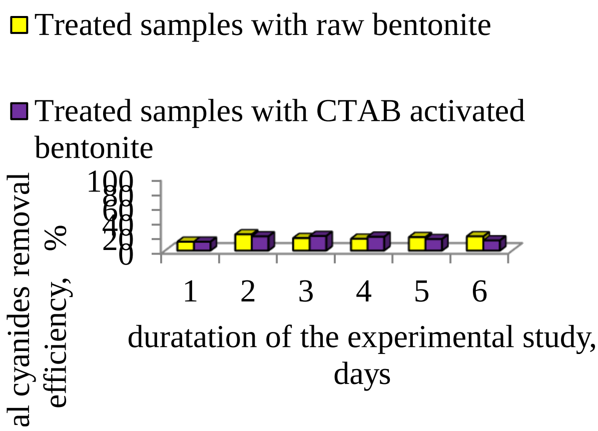

Figure 6. Total cyanides removal efficiency, % depending on the duration of the experimental study, days

The use of raw bentonite for the removal of phenolic compounds from ammonia-phenolic wastewater did not yield any significant results. Therefore, it can be concluded that raw bentonite is not an effective adsorbent for the removal of phenolic compounds from wastewater. However, with the use of CTAB activated bentonite, significantly better results were obtained in the removal of these ions present in ammonia-phenol wastewater. Which also contradicts the experimental study of Cao et al. 2013 that CTAB activated bentonite removes up to 81.36% of phenolic compounds from wastewater [41]. The highest percentage of removal was on 1st day of the experimental study and was 42.50%, while the lowest percentage of removal was 15.64%. The use of CTAB activated bentonite improved its adsorption characteristics, which is also confirmed by these experimental results. Al-Asheh et. al 2003 reported that using CTAB activated bentonite achieved better removal of phenolic compounds from wastewater than natural bentonite [42]. Wang et. al 2013 for the removal of phenol from wastewater with CTAB activated bentonite supported by $KMnO_4$ obtained the results that the removal efficiency was 92% [43]. **Figure 6.** shows the efficiency of total cyanides removal, % depending on the duration of the experimental study, in days.

The efficiency of cyanide removal from ammonia-phenol wastewater using raw and CTAB-activated bentonite did not show any significant differences in the results. With both adsorbents, the removal efficiency ranged from 12.12 mg/L on the 1st day of the experimental study to a maximum of 19.44 mg/L for raw bentonite, or up to 20 mg/L for CTAB activated bentonite, respectively. RezaeiKahkha et al. 2017, used activated bentonite to remove phosphate, nitrate and cyanide, where they achieved the maximum percentage of removal of these anions [44].

IV.CONCLUSION

In the treatment of ammonia-phenolic wastewater, CTAB proved to be a good adsorbent for the removal of total ammonia and phenol compared to raw bentonite. For the removal of cyanide ions, raw benonite is recommended. Although both adsorbents give similar percentages of removal, raw bentonite is considered a cheaper variant due to additional costs and time, compared to the activated one, so it would be the choice for removing these ions.

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