Desalination and Water Treatment
www.deswater.com

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doi: 10.1080/19443994.2012.708547

50 (2012) 87–94
December

Taylor & Francis
Taylor & Francis Group

Valorization of carbide lime waste, a by-product of acetylene manufacture, in wastewater treatment

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Received 11 January 2012; Accepted 8 May 2012

ABSTRACT

Every year increasing amounts of industrial waste are generated worldwide. Depending on their characteristics, wastes can represent an important source of secondary raw materials in order to replace natural resources. The growing amount of carbide lime waste (CLW), a by-product of acetylene production, has resulted in environmental problems. In the present study, the potential use of CLW for wastewater treatment was investigated. The main characteristics of the CLW were determined. Chemical and X-ray diffraction analyses indicated that CLW was similar in chemical and mineralogical compositions to industrial lime, except for the presence of carbon in the waste. Morphological and elemental chemical analyses by scanning electron microscopy and energy dispersive X-ray spectrometry revealed that CLW particles differ from industrial lime by the presence of carbon formations. The use of CLW was evaluated in the treatment of Annaba city wastewater effluent. CLW was found to be the suitable for the treatment of Annaba city wastewater for an optimal dose of $850\,\mathrm{mg\,L^{-1}}$. Percentage removal efficiency for turbidity, total suspended solids (TSS), chemical oxygen demand (COD) and 5-day biochemical oxygen demand (BOD_5) was found to be 96, 98.2, 90 and 84.5%, respectively. Residual turbidity in supernatant was 4.5 NTU and total residual bacteria was $68\,\mathrm{CFU\,mL^{-1}}$. Algerian effluent quality standards for TSS and COD were met after treatment. However, BOD_5, bacterial level and pH were high, emphasizing the need of pH adjustment and secondary treatment for the Annaba city effluent. The precipitation of heavy metals with CLW has been shown to be successful in reducing the level of soluble heavy metals in aqueous solution. The removal of heavy metals was enhanced at pH ranges 10−11 for zinc, 9.2−11.6 for lead, 4−11.8 for iron and 7−11.8 for copper. The results revealed that CLW can be effectively used in wastewater treatment.

Keywords: Valorization; Carbide lime waste; Acetylene production; Wastewater treatment

1. Introduction

Large quantities of industrial by-products are produced every year in Algeria, and a significant portion of these by-products continues to be landfilled as solid waste. Depending on their characteristics, wastes can represent an important source of secondary raw materials in order to replace natural resources. The growing amount of carbide lime waste (CLW), a by-product of acetylene production, has resulted in an environmental problem.

One of the conventional methods for producing acetylene ($CH{\equiv}CH$) is the action of water on calcium carbide. Calcium carbide (CaC_2) is produced in an electric furnace by heating a mixture of lime and

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carbonaceous materials such as coke, coal or charcoal. The calcium carbonate is first converted into calcium oxide and the coal into coke, and then the two are reacted with each other to form calcium carbide and carbon monoxide (Reaction (1)).

$$CaO + 3C \rightarrow CaC_2 + CO \tag{1}$$

Calcium carbide (or calcium acetylide) and water are then reacted to produce acetylene and lime slurry (Reaction (2)) [1,2].

$$CaC_2 + 2H_2O \rightarrow C_2H_2 + Ca(OH)_2 + 64\ \mathrm{kcal} \tag{2}$$

In acetylene manufacturing process, the lime slurry is pumped from the reactor to settling tanks where water is removed for recycling and sediment formed. Water from the slurry is saturated with acetylene and is therefore preferred as a source of water in the reactor. By using recycled water, the overall efficiency of the reaction is increased. Once water has been removed from the slurry for recycling, the sediment formed in the holding tanks is stockpiled on site or landfilled [3].

CLW, referred to as carbide lime sludge, hydrated lime waste, calcium hydroxide waste and other such designations, is generated as an aqueous slurry and is composed essentially by calcium hydroxide ($Ca(OH)_2 \approx 85–95\%$) with minor parts of calcium carbonate ($CaCO_3 \approx 1–10\%$), unreacted carbon and silicates (1–3%) [1,2,4–6]. The characteristics of the sludge are influenced by processing parameters during acetylene fabrication [1]. Although not being classified as dangerous/hazardous, its managing and disposal require special caution. Ammonium hydroxide present in supernatant (100–300 ppm) and acetylene dissolved in the water fraction may also be an issue, requiring appropriate ventilation during storage [2,4,5].

Carbide lime can be considered either as waste, affecting the environment, or as a resource when an appropriate valorization technology is implemented. The CLW may be used as a substitute for lime for agricultural purposes, civil constructions and several industrial processes [1,2,6–8]. However, it appeared to be suitable for wastewater treatment. This by-product was tested in order to introduce the CLW in the field of wastewater treatment and to increase its application in the field of environmental protection.

The originality of the present work is to demonstrate the possibility of using CLW in wastewater treatment. The widespread use of this by-product in wastewater treatment would ensure a very large market, and allows simultaneous dual action favourable to the environment: elimination of industrial waste and wastewater treatment. Additionally, it is necessary that the by-product leads to acceptable performance and economically attractive implementation.

Wastewater treatment in Algeria is evolving. Application of secondary biological treatment to industrial and municipal wastewaters is not widely practised due to a number of reasons which include high capital costs, lack of operation and maintenance skills and the absence of stringent enforcement of environmental standards [9–13]. Coagulation–flocculation–sedimentation, normally referred to as chemically assisted primary sedimentation or chemically enhanced primary treatment (CEPT), is a technology that appears to have potential in Algeria to cope with evolutionary demand of environmental protection.

In the present work, the main characteristics of CLW were determined, discussed and compared with those of limes used in water treatment. The objective of this study was to examine the treatability of Annaba city wastewater (Bouhamra River) by CLW using a conventional Jar test. Jar test is a valuable tool that can be used to evaluate the efficiency of a physical–chemical treatment. In addition, chemical treatment using CLW was investigated in terms of its capacity to remove heavy metals from contaminated water.

2. Experimental

2.1. Carbide lime waste

CLW samples for the present research were collected from the Entreprise Nationale des Gaz Industriels (ENGI) situated in El-Hadjar, Annaba (Algeria). These samples were completely dried in an oven and the obtained powder was stored in a desiccator until use. The obtained material has a grayish colour and is fine in particle size.

The particle size analysis of the CLW was determined by passing the representative samples through a series of various opening size sieves, followed by weighing the size fractions.

The morphology of the CLW particles was observed by scanning electron microscopy (SEM), using a LEO 440 microscope. A Kevex Sigma Energy Dispersive System (X-ray spectrometer), coupled to the microscope, was used for elemental chemical analyses of the samples.

The crystal structure of the CLW samples was analysed on a powder X-ray diffractometer Bruker D8-Advance using Ni-filtered Cu Kα radiation. The diffractogram was taken with a step of 0.02° using 2 s counting time.

Table 1
Wastewater quality parameters for raw homogenized Annaba city wastewater (Bouhamra River)

Parameter	Minimum	Maximum	Mean
Turbidity (NTU)	55	230	130
pH	7,2	8,3	7,5
Conductivity ($\mu S\,cm^{-1}$)	1,130	1,370	1,235
TSS ($mg\,L^{-1}$)	229	1,187	541
COD ($mg\,L^{-1}$)	437	1,432	960
BOD_5 ($mg\,L^{-1}$)	59	268	176
Number of bacteria ($CFU\,mL^{-1}$)	23×10^5	117×10^8	236×10^6

2.2. *Annaba city wastewater*

Raw wastewater samples were collected from Bouhamra River situated in Annaba, Algeria. This much polluted river gathers wastewater of Annaba city. Various quality parameters of wastewater were determined according to the procedures laid down in the Standard Methods [14] and are summarized in Table 1.

2.3. *Jar test methodology*

The experimental set-up used for the coagulation–flocculation experiments at laboratory scale consisted of a Jar-test device (WTW, Germany) in which six stirring blades were connected to a motor that operated under adjustable conditions. The system permitted the experiments to be performed with ease. In this study, standardized mixing speeds and durations were used which are reported in the literature [15–17].

For each jar test, 500 mL of wastewater was taken in the jars and rapidly mixed at 200 rpm for 3 min. The required doses of waste lime were added to the jars during the rapid mixing. The rapid mix was followed by tapered flocculation at 40 rpm for 25 min. Afterwards a settling time of 30 min was given before drawing the sample. Jar tests were performed using raw homogenized wastewater without pre-settling.

2.4. *Analytical procedures*

The influent and effluent quality parameters including pH, turbidity, total suspended solids (TSS), conductivity, chemical oxygen demand (COD) and 5-day biochemical oxygen demand (BOD_5) were determined according to Standard Methods [14]. The parameters are given in Table 2.

Table 2
List of analysed parameters and adopted analytical procedures

Parameter	Reference method
Turbidity	SM 2130B
pH	SM 4500-H^+ B
Conductivity	SM 2510B
TSS	SM 2540D
COD	SM 5220D
BOD_5	SM 5210B

The number of viable bacterial cells was determined as colony forming units (CFU). 1 mL of supernatant was serially diluted (10−1 to 10−9) and volumes of 0.1 mL were aseptically inoculated onto nutrient agar (spread-plate method). Following incubation at 30 ± 0.1 °C for 24 h the number of colonies was enumerated and the amount of free-living bacteria in the process water (expressed in CFU/mL) was determined.

2.5. *Precipitation of heavy metals*

Solutions (synthetic effluents) containing Cu(II), Cr (III), Pb(II) and Zn(II) were prepared from their respective standard reagent-grade metal nitrate salts. Each metal salt, $Cu(NO_3)_2 \cdot 3H_2O$, $Zn(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_2 \cdot 9H_2O$ and $Pb(NO_3)_2$, was dissolved in distilled water at a concentration of $200\,mg\,L^{-1}$ for precipitation studies.

The precipitation of heavy metals from the synthetic effluents involved batch experiments at 20 °C in sealed 500 mL vessels. After the addition of CLW, the solutions were agitated at 200 rpm for 3 min using a mechanic stirrer, followed by gentle stirring at 40 rpm for 7 min to promote coagulation and agglomeration [18]. After mixing, the suspensions were allowed to settle for 30 min, and the supernatant from each vessel was collected and filtered through 0.45 μm Whatman filters and analysed by atomic absorption spectroscopy (Perkin−Elmer A310).

3. Results and discussion

3.1. *Characterization of CLW*

The particle size distribution of CLW is shown in Fig. 1. This figure indicates that the CLW is extremely fine in size. From the results of Fig. 1, the effective diameter (D_{10}), mean particle size (D_{50}) and uniformity coefficient (D_{60}/D_{10}) of CLW were 75 μm, 118 μm and 2.1, respectively. This indicates that 90% of CLW particles passed through the 75-μm sieve.

The results of the chemical analysis of the CLW determined by X-ray fluorescence technique (Model

S4 Explorer 7KP103, Brucker) are shown in Table 3. It is clear that the CLW is a calcium-based lime since it contains 67.03% by weight of calcium oxide, and no magnesium. The presence of 0.12% sulfur may be the cause of the waste's grey color. In comparison with commercial limes [2], CLW has higher content of SiO_2, Al_2O_3 and Fe_2O_3 and lower amount of CO_2.

The X-ray diffractogram of the CLW (Fig. 2) only revealed the presence of calcite $CaCO_3$ (JCPDS 05-0586) as crystalline phase. The presence of calcite explained the high loss on ignition (24.32%), observed in the chemical analysis (Table 3).

SEM image of CLW in Fig. 3 discloses relatively heterogeneous surfaces. The surface of the aggregates presents an irregular texture. According to the energy dispersive X-ray spectrometry (EDS) spectrogram in Fig. 4(A), light-grey area all over the agglomerate accounts for calcium hydroxide clusters with traces of carbon. The dark-grey material indicated the presence of Ca, Al, Si, C, S and O (Fig. 4(B)).

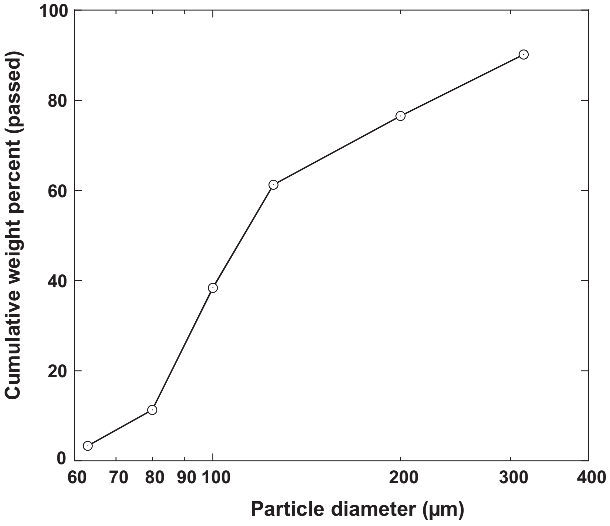

Fig. 1. Particle size distribution of CLW.

Table 3
Chemical analysis of the CLW

Parameter	Weight percentage
CaO	67.03
MgO	0.00
SiO_2	2.06
Al_2O_3	2.14
Fe_2O_3	2.40
SO_3	0.12
Residual CO_2	1.28
C	0.65
Loss on ignition	24.32

Based on the characterization results, the CLW has the potential to be used in wastewater treatment. Nevertheless, the use of the recycled material as a chemical for CEPT of Annaba city wastewater and for precipitation of heavy metals in industrial effluents requires further research to provide reliable conclusions regarding its in-use performance.

3.2. Treatment of Annaba city wastewater

Coagulation–flocculation is an essential unit process in removing colloidal particles and organic matters in wastewater treatment. The process was found to be cost effective, easy to operate and uses less energy than alternative treatment.

The effect of CLW dosage on turbidity removal is shown in Fig. 5. It is evident from the figure that turbidity removal was enhanced by increasing CLW dose in the range 400–850 $mg\,L^{-1}$. A maximum removal of 96% with respect to raw homogenized wastewater occurred at a CLW dose of 850 $mg\,L^{-1}$. The effluent

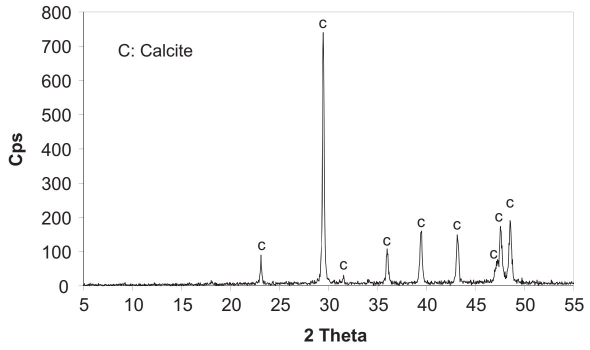

Fig. 2. X-ray diffractogram of CLW.

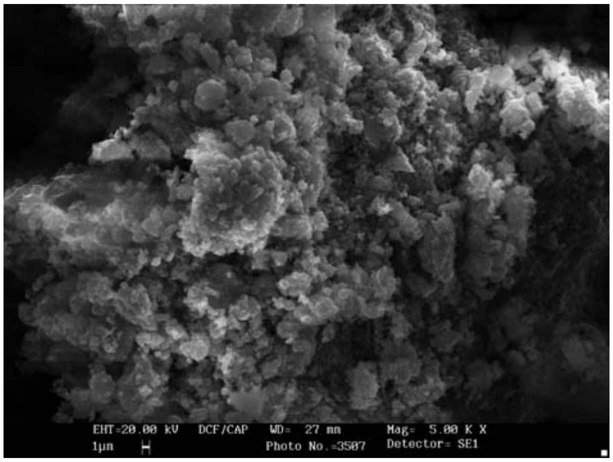

Fig. 3. SEM image of CLW.

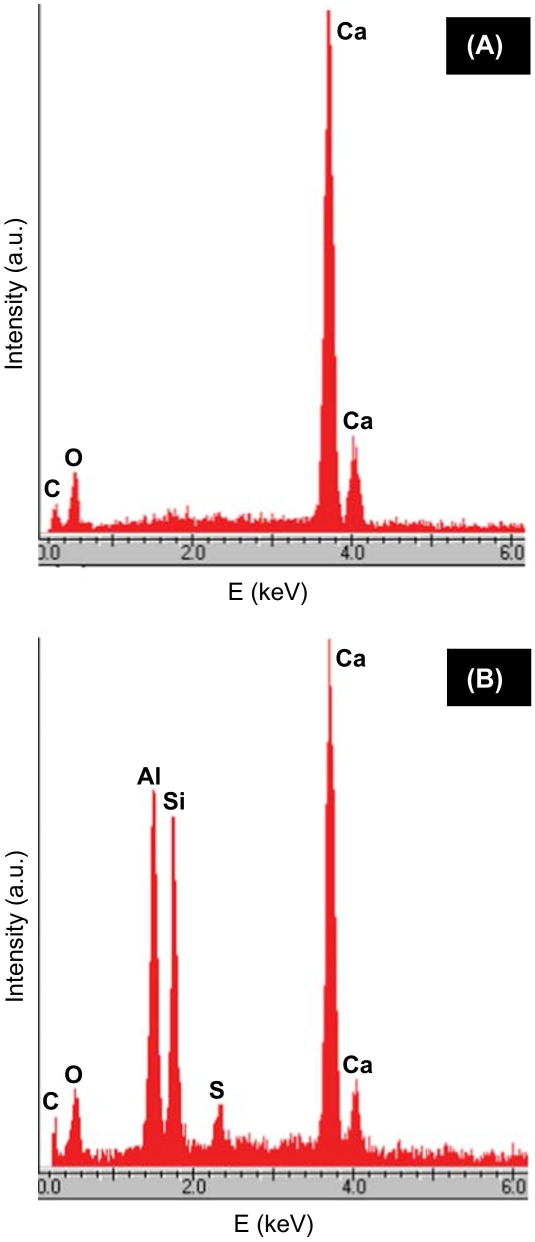

Fig. 4. EDS analysis of CLW.

turbidity at this dose was 4.5 NTU. At low CLW doses, a bad removal of turbidity was noticed. This zone corresponds to the concentration range where poor floc formation was observed. Although the destabilization of colloidal particles by CLW was fast and occurs before precipitate formation, there was no settling. Probably under these conditions, though there was neutralization of negative charges on colloids, much negative charges would prevent floc formation. At CLW doses above 850 mg L^{-1}, residual turbidity slightly increased.

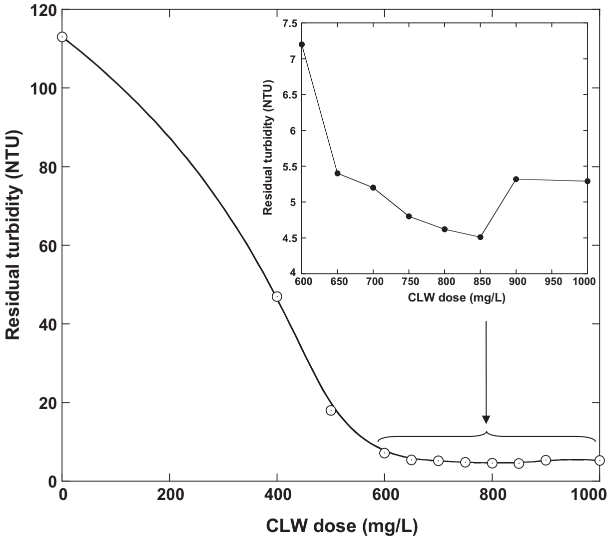

Fig. 5. Effect of CLW dose on residual turbidty.

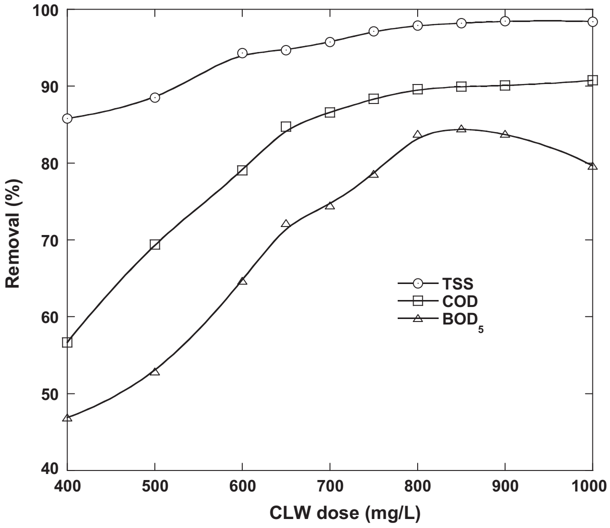

Fig. 6. Influence of CLW dose on the removal of TSS, COD and BOD_5.

It can be observed from Fig. 6 that a maximum removal of TSS occurred at CLW doses above 850 mg L^{-1}. At these doses, removal of TSS was $\geqslant$98%. No further appreciable reduction in TSS could be observed at higher dose of CLW. At a CLW doses of 850 mg L^{-1}, TSS of treated effluent was 13.3 mg L^{-1}. The effluent standard in Algeria for TSS is 35 mg L^{-1} [19]. Thus, CEPT at the optimum dose of CLW was successful in meeting effluent standards for TSS.

The residual COD in relation with the CLW dose was represented in Fig. 6. It appeared that the COD removal increased proportionally with the increasing in the amount of the CLW. COD abatement was enhanced rapidly during the CLW dosages of 400–800 mg L^{-1}. However, when CLW dosage exceeded 850 mg L^{-1}, COD removal efficiency was not enhanced obviously and remained at 89.6–90.8%. The effluent COD value at CLW dose of 850 mg L^{-1} was 63.2 mg L^{-1}. It seems that the resulted treatment is sufficient to discharge the wastewater because the effluent standard in Algeria for COD is 120 mg L^{-1} [19].

BOD_5 removal efficiency as a function of CLW dose is given in Fig. 6. The removal efficiency first rises with increasing CLW dose, reaching the maximal value, and then slightly decreases with further carbide lime addition. Maximum percentage removal of 84.5% for BOD_5 could be observed at a CLW dose of 850 mg L^{-1} that corresponds to a residual BOD_5 of 40 mg L^{-1}. The effluent standard in Algeria for BOD_5 is 35 mg L^{-1} [19]. Consequently, the resulted treatment at the optimum dose of CLW was not successful in meeting effluent standard for BOD_5.

Because the TSS and COD removal efficiencies increase by increasing carbide lime dose, the optimum CLW dose in this study is defined as the lowest dosage at which maximum removal efficiency for turbidity and BOD_5 was achieved. CLW dosage corresponding to the lowest residual turbidity coincides with the higher BOD_5 removal efficiency. Thus, the optimum dose of CLW was 850 mg L^{-1}.

The effect of CLW dosage on bacteria removal is shown in Fig. 7. Removal efficiency was increased with incrementing CLW addition. Total residual bacteria were 68 and 28 CFU mL^{-1} For CLW doses of 850 and 1,000 mg L^{-1}, respectively. Concerning microbiological standards in wastewater disposal and reuse, the number of faecal coliforms or streptococci, according to the United States Environmental Protection Agency, must be <10 CFU mL^{-1} [20], which is also in accordance with the World Health Organization [21] recommendation. Consequently, the microbiological standards are not reached.

The influence of the CLW amount on the pH and electrical conductivity of the resulting effluent was shown in Fig. 8. It was observed that the pH and electrical conductivity increased with increasing in the amount of CLW. After treatment using optimum dosage of CLW (850 mg L^{-1}), pH and electrical conductivity of the resulted effluent were 11.5 and 1,872 μS cm^{-1}, respectively. The effluent standard in Algeria for pH is in the range 6.5–8.5 [19]. Consequently, BOD_5, bacterial level and pH of the effluent from CEPT (850 mg L^{-1}) were still higher than the effluent standards thus emphasizing the need of pH adjustment and secondary treatment.

3.3. Removal of heavy metals from contaminated water

Chemical precipitation is the most widely used conventional process for heavy metal removal from inorganic effluent. The conceptual mechanism of heavy metal removal by chemical precipitation is presented in Eq. (3).

$$M^{2+} + 2OH^{-} \rightarrow M(OH)_2 \downarrow \tag{3}$$

where M^{2+} and OH^{-} represent the dissolved metal ions and the precipitant, respectively, while $M(OH)_2$ is the insoluble metal hydroxide. Adjustment of pH to

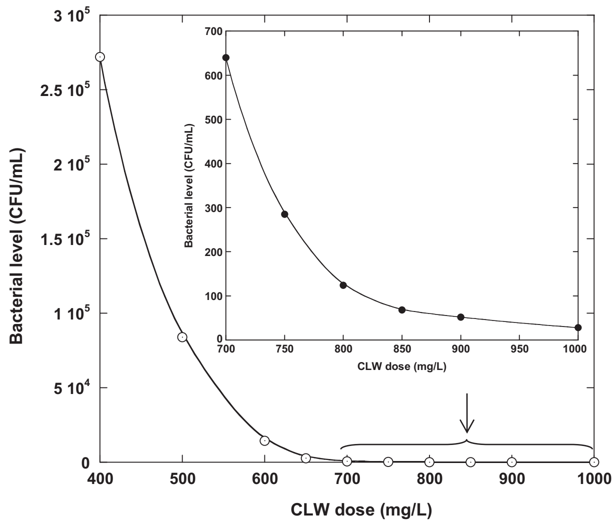

Fig. 7. Effect of CLW dose on residual bacterial level.

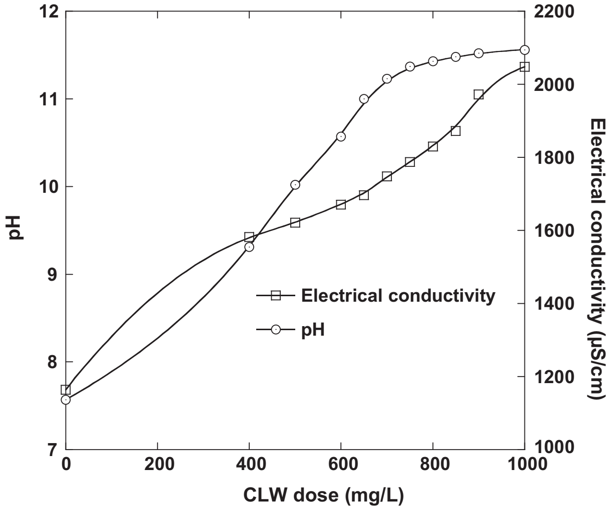

Fig. 8. Evolution of pH and electrical conductivity vs. CLW dose.

the basic conditions is the major parameter that significantly improves heavy metal removal by chemical precipitation. Lime and limestone are the most commonly employed precipitant agents [22–24]. Lime precipitation can be employed to effectively treat inorganic effluent with a metal concentration of higher than 1,000 mg L^{-1}. Other advantages of using lime precipitation include the simplicity of the process, inexpensive equipment requirement and convenient and safe operations. However, chemical precipitation requires a large amount of chemicals to reduce metals to an acceptable level for discharge. Consequently, in the present work, the potential use of CLW for chemical precipitation of heavy metals was investigated.

Fig. 9 shows the dependence of heavy metals precipitation on solution pH, which was adjusted by CLW additions. Of the four heavy metal ions of interest, the removal of iron was the greatest and in the decreasing sequence: iron, copper, lead and zinc. Copper was removed at a broader pH range. At pH 3, 42.2% of iron was precipitated, increasing to 100% in pH range 9−11. At pH 11.8, iron precipitation efficiency is slightly decreased (99.4%). The precipitation of copper increased rapidly with pH and 92.7% was removed between pHs 6 and 7. No further appreciable reduction in copper concentration could be observed at higher pHs (7−11.8). When the pH increases from 7 to 11.8, copper removal efficiency slightly increases from 95.8 to 98.7%, respectively. For lead, 82.6% removal occurred between pHs 5 and 11. The optimum pH for lead precipitation using CLW was 11, with a removal efficiency of 90.5%. At pH 11.6, lead removal decreased to 81.4%. For zinc, it can be noticed that the precipitation efficiency increased with an increase in pH up to 10.4. At pH 6, 3.9% of zinc was precipitated, increasing to 59.2% at pH 10.4. In the pH range 10.4−11.8, zinc removal efficiency decreased from 59.2 to 35.8%, respectively.

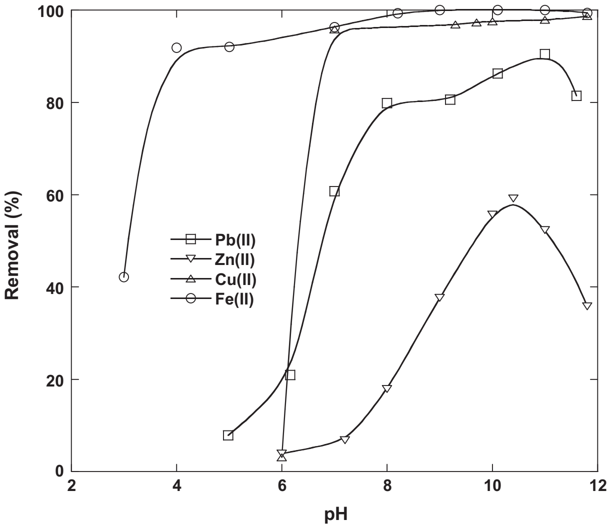

Fig. 9. Dependence of heavy metals' precipitation on solution pH.

The results of this investigation have shown that CLW can be readily utilized as a replacement to conventional lime used for wastewater treatment.

4. Conclusions

This study shows that CLW can be successfully used in wastewater treatment. CLW characterization indicates that the CLW is extremely fine in size (mean particle size of 118 μm). Chemical and X-ray diffraction analyses indicated that CLW was similar in chemical and mineralogical compositions to industrial lime, except for the presence of carbon in the waste. Morphological and elemental chemical analyses by SEM and EDS revealed that CLW particles differ from industrial lime by the presence of carbon formations. Based on the characterization results, the carbide lime has the potential to be used in wastewater treatment.

CLW was found to be the suitable for the treatment of Annaba city wastewater for an optimal dose of 850 mg L^{-1}. The precipitation of heavy metals with CLW has been shown to be successful in reducing the level of soluble heavy metals in aqueous media. The results revealed that CLW can be effectively used in wastewater treatment.

Acknowledgements

The financial support by the General Directorate for Scientific Research and Technological Development (PNR project No. 4/D/25) and the Ministry of Higher Education and Scientific Research of Algeria (project No. J0101120090018) is greatly acknowledged.

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