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BARIUM RECOVERY FROM SLAG USING ULTRASONICATION

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Abstract

Ultrasonication is a process which has high potential in the processing of liquids and slurries by improving the mixing and chemical reactions. Its main application is its efficiency in direct degradation of pollutants, dehydration of sludge, slurry filtration and metal recovery. Barium is a toxic radionuclide released during industrial activities which leads to environmental pollution. In the current study, recently developed methods such as ultrasonication and microwave are discussed which aids in the recovery of barium from industrial slag. Power ultrasound is done at 20 kHz using power ranging from 200W to 2000W. Microwave treatment method is mainly adopted at two different temperatures of 160° C and 360°C. The process is carried out using four different acids with normalities ranging from 0.1 to 0.5N at various timings and the effect of time and normalities are studied.

Keywords: Ultrasonication, Microwave, metal recovery**.**

Introduction

Barium is an alkaline earth element which occurs as a trace metal in igneous and sedimentary rocks. In nature it occurs principally in combined states as barite (BaSO4) and witherite (BaCO3). Barium is used industrially in a variety of forms. Barium causes environmental pollution when it is released through industrial activities. They dissolve easily in water and are found in lakes, rivers and streams. Barium compounds when dissolved in water can cause many harmful effects on human health. Because of their water solubility these barium compounds can spread over larger distances. When fishes and other aquatic organisms absorbs barium compounds, barium will accumulate in their bodies. Since it forms insoluble salts with other components in the environment, such as carbonate and sulphate it causes great risk. Smallamount of water soluble barium causes human health problems like breathing difficulties, increased blood pressure, stomach irritation, muscle weakness, changes in nerve reflexes, swelling of brains and liver, kidney and heart damage etc. All barium compounds that are water or acid soluble are poisonous.

Heavy metals are used in industry and are consequently discharged into the environment. These metals will accumulate in soils and causes environmental pollution. Thus many technologies are currently used to clean up heavy metal contaminated soils. Therefore, many treatment technologies like pump-and-treat, cut-off wall, soil washing, soil flushing, soil vapor extraction, bioremediation, electrokinetics, ultrasonication etc have been used. Of these, electrokinetic remediation and ultrasonic remediation are good for the remediation of contaminated soil. The importance of environmental pollution control has been significantly increased in the recent

years and therefore many, pretreatment process has been developed to to improve the sludge treatment. It mainly includes ozone pretreatment, ultrasound, ball mill and thermal treatment.

Ultrasonication is an effective pretreatment method which is used to enhance the biodegradability of the sludge. This process disrupts the physical, chemical and biological properties of the sludge. It is also important in the wastewater treatment plants in order to treat and dispose off the sludge. Different factors affect the degree of disintegration. These include the
sonication parameters and the sludge characteristics. When asymmetric crystals are compressed, they generate electric energy. This is known as piezoelectric effect. But, when electrical current
is passed through the crystals they contract. When the electrical current is alternated through the crystals, they are contracted and expanded creating a mechanical vibration. Here, motors are not required to convert the electrical energy into mechanical energy. This property is utilised by the process of ultrasonication where high frequency oscillating mechanical vibrations are created by stimulation with high frequency oscillating electric currents. The instrument used for ultrasonication is a sonicator. The standard laboratory sonicators run at a frequency of 20 kHz to 23 kHz. These instruments take the alternating current and magnify or convert the cycles upwards. The piezoelectric crystals are stimulated by the incoming electrical current which is converted to a high frequency current. A probe is present, to which the crystals are attached here.

This will be immersed into the liquid. Vibrational energy is imparted to the into the liquid. Cavities are created since the flow of the liquids is not fast. The expansion of the crystals leads to he creation of microscopic shock waves. The process is known as cavitation and it is extremely powerful. The cavities form and collapse in microseconds releasing a huge amount of energy in the liquid. An important factor in sonication is the size of the particles.

Ultrasonication associates with two important phenomenons such as formation and collapse of cavitation bubbles that generates extremely high pressures and temperatures in the center of cavitation bubbles. It is new and a clean field due to the limitation of available methods using no chemicals for the elimination of undesirable chemicals from the contaminated matrix. Ultrasonication was used as the pre-treatment process to improve waste water and saline solution disinfection. Ultrasound irradiation could also enhance membrane filtration of waste water and sludge stabilization. Ultrasonication exhibits a great potential of being environmental friendly and economically competitive treatment method. In the current study, an attempt has been made to find out the effective method of barium removal by comparing ultrasonication and microwave methods at various temperatures by using different acids with varying normalities. Barium carbonate solution is used as the stock solution for analysing barium in the atomic absorption spectrophotometer. It is also known as witherite and used as a component in rat poisons, bricks, glazes and cement. It is made from barium sulphide on treatment with sodium carbonate at 60-70°C or by passing carbon dioxide at 40 to 90°C. It reacts with hydrochloric acid to form soluble barium salts such a s barium chloride. Barium sulphate is formed on reaction with sulphuric acid.

MATERIALS AND METHODS

The heavy metal slag was collected from an industry in Ranipet, Tamil nadu. It is then crushed into very fine powder and then sieved through a 150µ sieve. This powder was then used for the further experiments.

Initially the calibration curve of barium was obtained. Barium carbonate solution of 1000mg/L was prepared by dissolving 1.438g barium carbonate in 20ml of hydrochloric acid and then diluted to 1L by using distilled water. By using this solution the respective standards were prepared varying from 100mg/L to 500mg/L. 100ml of this solution gave a 100mg/L solution; 200ml gave a 200mg/L

Volume: 1 Issue: 1 08-Jun-2013,ISSN_NO: 2320-7256

solution and so on up to 500mg/L. These standards were then analysed using AAS at a wavelength of 350.1nm and the corresponding absorbance was obtained. The absorbance is then plotted against concentration and the standard calibration curve was obtained. This curve can be used for finding out the concentration of the samples.

The solutions of various acids were prepared like hydrochloric acid, sulphuric acid, acetic acid and nitric acid with the normalities ranging from 0.1 to 0.5 N. Acid extraction was done by adding 8g barium slag to 40ml of each of the acids, i.e. 1:5 ratios. These samples were then kept in a shaking incubator for 24 hours at 30 rpm. The samples were then filtered and analysis was done by first diluting the samples to a lower concentration.

The sonication is done at a frequency of 20 kHz and supplied with an adjustable power of 200-2000W. Different acids were subjected to ultrasonication at different timings, i.e., 5, 10, 15 and 30 minutes at various normalities. After sonication centrifugation is done at 5000rpm for 30 minutes. The centrifuged samples were then filtered using Whatmann filter paper. The samples were diluted before analysis due to the high concentration of Barium. Without any dilution, the dilution factor was 5 since the slag and acid were taken in a 1:5 ratio. Dilutions were done by initially pipetting out 1ml from the filtered samples and making it upto 100ml. After 1 dilution, the dilution factor became 5x100. If the concentration still didn't fall in the preferred range, it had to be further diluted by pipetting out 1ml from the already diluted sample and making it upto 100ml. The dilution factor after this dilution would be 5x100x100. This dilution had to be carried on till the concentrations in the preferred range were obtained. The dilution factor should be
multiplied with this concentration to obtain the original concentrations of the sample after ultrasonication. The samples were analysed in the AAS in order to find out the absorbance. AAS is the atomic absorption spectrophotometer. It follows the principle of the Beer Lambert Law. For analysing barium, the Direct Nitrous Oxide Acetylene Flame Method is used.

RESULTS AND DISCUSSIONS

The standards of barium carbonate varying between 100 – 500mg/L were prepared and these were analysed in the AAS. The wavelength used was 350.1nm. The absorbance was obtained as follows

Concentration(mg/L)	Mean absorbance
100	0.0046
200	0.0099
300	0.0159
400	0.0214
500	0.0276

Using this known concentration and the obtained absorbance the calibration curve for barium was plotted. This curve could be further used for finding out the concentrations of the unknown samples.

Volume: 1 Issue: 1 08-Jun-2013,ISSN_NO: 2320-7256

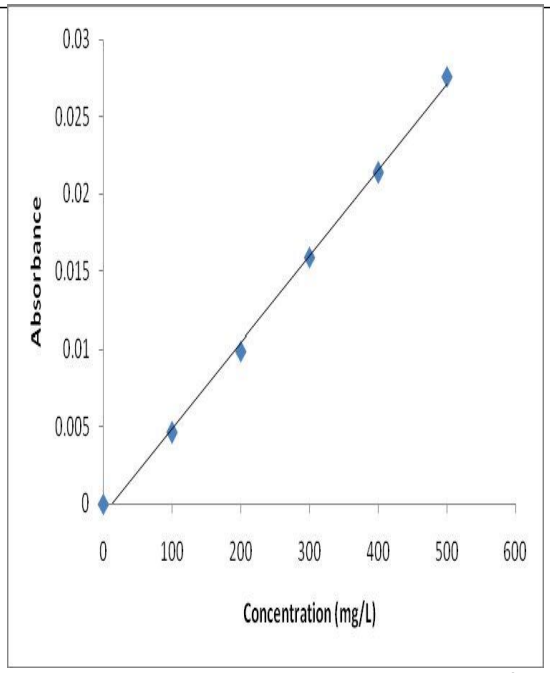

Fig 1: Standard calibration curve for Barium

Ultrasonication was carried out for acids of varying normalities for different times. These sonicated samples were then centrifuged and filtered before analysis. The samples to be analysed were diluted 2 fold since barium is present in very high concentrations. Ultrasonication was carried out for 5, 10 and 15 minutes and the concentration of the samples were analysed.

Acid	Time for sonication (minutes)	Absorbance	Concentration(g/L) Dilution Factor = 5x10000
0.1N HCl	5	0.0011	8500
0.1N HCl	10	0.0027	16500
0.N HCl	15	0.0025	18500

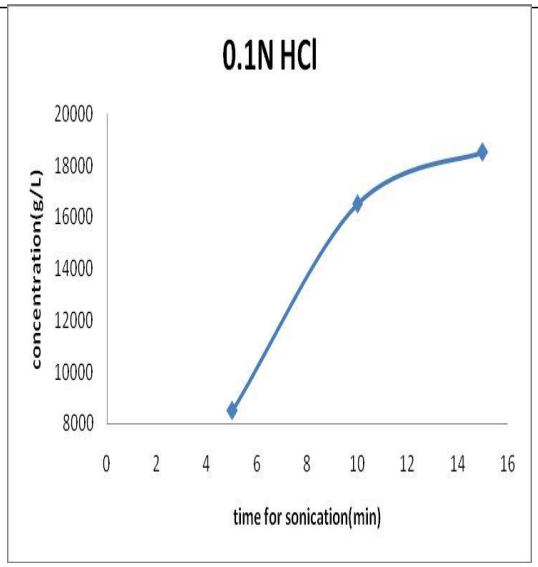

Fig 2: Concentration Vs time for sonication for 0.1N HCl

From the graph, it is observed that the maximum removal was obtained at a minimum sonication time and as the time increased the concentration further increased. The lowest concentration of 8500 g/L was observed for a sonication time of 5 minutes.

Acid	Time for sonication (minutes)	Absorbance	Concentration(g/L) Dilution Factor = 5x10000
0.2N HCl	5	0.0026	18000
0.2N HCl	10	0.0024	19000
0.2N HCl	15	0.0030	21500

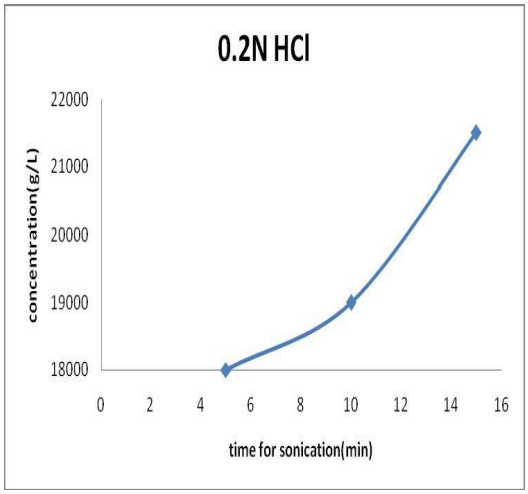

Fig 3: Concentration Vs time for sonication for 0.2N HCl

For 0.2N HCl also, the same trend was followed. The minimum concentration was observed for the least sonication time, i.e 5 minutes. This was about 18000g/L. As the sonication time increased the concentration of Barium in the solution increased giving a maximum for 15 minutes sonication time.

At 5 minutes sonication time, the concentration of 0.1N HCl was 8500/L but for the same parameters the concentration of 0.2N HCl was observed to be 18000g/L which showed that theremoval efficiency decreased with an increase in normality and also with increase in sonication time.

CONCLUSION

Normality of the acids and the sonication time played an important role in the removal of barium from the slag. Increase in normality leads to a decrease in removal efficiency even for the same sonication time. Also, maximum removal occurs at a lower sonication time. As the sonication time increases the concentration also increased.

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