CHARACTERIZATION OF SILICA FROM CLAY MINERALS IN KYAUKPADAUNG TOWNSHIP AND ITS APPLICATION ON LEAD CONTAMINANT WASTE WATER

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Abstract

In this research, the clay sample was collected from Khin Phone Chone Village, Kyaukpadaung Township, Mandalay Region. Elemental Analysis of the clay sample was determined by EDXRF technique. The extraction of silica from sample was performed by using various concentration of NaOH and HCl, 2 M, 4 M, 6 M, 8 M and 10 M. Then, silica was prepared from the sample using various stirring time and different temperature. For the sample, stirring time 60 min, 90 min and 120 min were used. By using different temperature such as 60 ℃, 80 ℃, 100 ℃, the optimum temperature for silica extraction was chosen at 80 ℃ for the sample. The extracted silica was characterized by using EDXRF, FT-IR, XRD and SEM analysis. From EDXRF data, the highest amount of silica were found to be 81.892%. From FT IR spectra, the peak at 1052 cm^{-1} for extracted silica was indicated the presence of Si-O stretching. SEM image showed that silica from clay has amorphous nature. The adsorption behavior of extracted silica has been studied. Waste water from lead acid battery industry were treated with extracted silica by using standing method. Before and after treatment, the quantities of waste water were determined. After treatment, the percent removal was found to be 64.76% from sample (1) and 34.84% for sample (2).

Keywords: Clay, Silica, EDXRF, FT IR, XRD, SEM analysis, standing method

Introduction

Clay minerals are the characteristic minerals of the earth's near surface environments. They form in soils and sediments, and by diagenetic and hydrothermal alteration of rocks. Water is essential for clay mineral formation and most clay minerals are described as hydrous alumino silicate. Structurally, the clay minerals are composed planes of cations, arranged in sheets, which may be tetrahedrally or octahedrally coordinated (with oxygen), which in turn are arranged into layers often described as 2:1 of they involve units composed of two tetrahedral and one octahedral sheet, or 1:1 of they involve units of alternating tetrahedral and octahedral sheets. Additionally some 2:1 clay minerals have inter layers sites between successive 2:1 units which may be occupied by interlayer cations, which are often hydrated. The planer structure of clay minerals give rise to characteristic platy habit of many and to prefect cleavages (Amonette, *et al*., 1994).

Clay minerals are included in several health care formulations. In particular, they are presented in many semisolid preparations with functions, including stabilization of suspensions and emulsions viscosizing and other special rheological tasks, adsorption of greases, control of heat release, etc (Barnhisel, *et al*., 1989).

Silica is another name for the chemical compound silicon dioxide. Each unit of silica includes one atom of silicon and two atoms of oxygen. Silica makes up the mineral called quartz and it is the most abundant mineral in the earth's crust. It is the main component of most sand and the primary ingredient in glass. Today, there are many industrials, fillers, electronics and water filtration. In this research work, clay mineral was used as precursor in silica extraction (Ghosh, *et al*., 2013).

Nowadays lead contamination in a environment is a very important problem of worldwide concerning due to its highly toxic and nonbiodegradable in nature. There are many ways that lead

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is released into aquatic system such as natural phenomenon, urban, agricultural and industrial activity.

Adsorption process is considered very effective in industrial wastewater treatment. It proves superior to the other processes by being sludge free and can completely remove even very minute amounts of heavy metal in wastewater. Adsorption process using silica is very effective for removal of heavy metal from wastewater but its high cost has provided the search for alternatives and low-cost adsorption (Sone, *et al*., 2009).

Materials and Methods

Sample Collection

Clay sample was collected from Khin Phone Chone Village, Kyaukpadaung Township, Mandalay Region. Afterwards, pieces of macroorganic matters were discarded the clay aggregates were broken up by grinding lightly in motor and pestle. Then, it was meshed through 40 mesh size sieve and the fine powder sample was stored for further uses.

Figure 1 Sample collection site of Kyaukpadaung Township
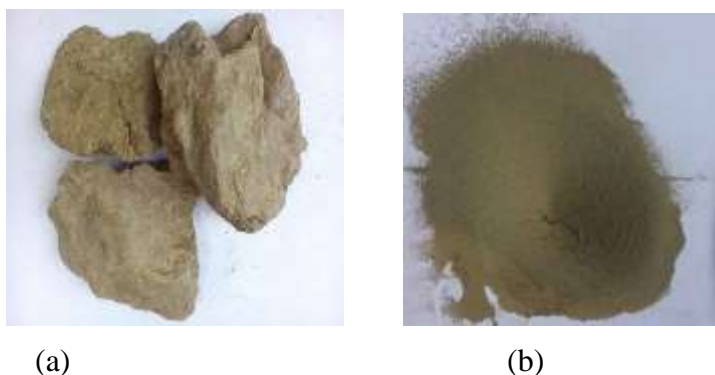
(a)   (b)

Figure 2 (a) and (b) Clay sample in Khin Phone Chone Village, Kyaukpadaung ownship

Determination of Mineral Content in Clay Mineral

The selected clay sample was measured at the Department of Physics, University of Mandalay by EDXRF technique.

Preparation of Silica from Clay Sample with Various Concentrations

(10 g) of sample was weighed accurately and placed in beaker. And then the sample was washed with 100 mL of distilled water and filtered by filter paper. After that, the residues were used for silica extraction 100 mL of 2 M NaOH was added to the residue in beaker. The beaker was covered for 60 min heated at 80 ℃ and constant stirring. The solution was filtered by filter paper and carbon residues were washed with 100 mL of boiling water. The filtrate and washing were allowed to cool at room temperature and were precipitated 2 M HCl with constant stirring to pH4. Silica gel started to precipitate when pH decreased to <10. The silica gel was aged for 18 h. This gel filtered by filter paper and dried at 80 ℃ for 12 h. Similarlly, 4 M, 6 M, 8 M and 10 M NaOH and HCl were used for above same procedures to prepare silica.

Preparation of Silica by Various Temperature

According to former procedure, silica powder was prepared by using various temperatures (60 ℃, 80º C and 100 ℃).

Preparation of Silica by Various Reaction Time

According to former procedure, silica powder was prepared by using various reaction times (60 min, 90 min and 120 min).

Characterization of Extracted Silica from Clay Mineral

Determination of Elemental Contents by EDXRF Technique

The oxide powder was determined by EDXRF analyzer (EDX-700 spectrometer) to examine the presence of elements in the powder. It was measured at Monywa University. The EDXRF spectrum result of oxide powder is shown on Table 5.

FT IR Analysis

Prepared pure silica sample was analyzed by FT IR spectrophotometer. The procedure was used in accordance with recommended standard procedure as reported in FT IR spectrophotometer catalogue. The characteristic feature of FT IR spectrum of prepared pure silica is show in Figure 7. The spectral assignment for prepared pure silica sample is also presented in Table 6.

XRD Analysis

The oxide powder was characterized by X-Ray Diffractometer (XRD 6000, Shimadzu, Japna) for phase analysis at the West Yangon University. The XRD pattern of silica is indicated in Figure 8.

SEM Analysis

The oxide powder was characterized by SEM (JEOL-JSM-5610 Series, Japan) analyzer to know the particle size and surface morphology of the powder. It was measured at the West Yangon University. The SEM microgram of oxide powder is indicated in Figure 9.

Determination of Removal Efficiency of Silica by Standing Method

(Column method)

Materials

Lead (II) nitrate

Sodium hydroxide solution (0.1 - 1.0 M)

Hydrochloric acid (0.1- 1.0 M)

Apparatus

Column, Measuring Cylinder, Beaker, Funnel, Filter Paper

Procedure

(5 g) of silica was weighed and placed in the column. 120 mL of mg/mL of lead aqueous solution was poured into the column containing silica. The lead solution was passed through the silica and the filtrate was collected at room temperature the flow rate was 15 drops per minute. After that, Pb^{2+} ion in the filtrate was determined by atomic adsorption spectrophotometer.

Sample Collection (Waste water)

The waste water sample was collected from Shwe Nan Taw Battery Factory, Industrial Zone (2), Mandalay Region. Two different site of waste water were collected. Sample (1) was collected from the wastewater tank of the industry and sample (II) was collected from pond behind the Shwe Nan Taw Battery Factory. The wastewater from the tank drains directly into this pond.

Treatment of Wastewater by Extracted Silica (Standing Method)

Materials

Industrial Wastewater

Apparatus

Column, Measuring Cylinder, Beaker, Filter Paper

Procedure

(5 g) of silica was weighed and placed in the column. 120 mL of waste water of was poured into the column containing silica. The industrial wastewater was passed through the silica and filtrate was collected at room temperature. The flow rate was 15 drops per minute. Before and after treatment, Pb^{2+} ion concentration was determined by atomic absorption spectrophotometer. Lead content in waste water (before and after treatment) is indicated in Table 7 and 8.

Results and Discussion

Mineral Contents of Clay Sample by EDXRF Analysis

The observed spectrum indicates that 11.850% Si, 4.826% Fe, 3.740% Al, 0.487% K and 0.434% Ca for clay sample. Silica is the rich mineral in clay sample. In this research work, clay sample was used as precursor in silica extraction. For qualitative determination of elements in clay sample, EDXRF spectrophotometer was used. The relative abundance of elements in clay sample is presented in Table 1.

Table 1 Relative Abundance of Mineral Contents in Clay Sample by EDXRF Analysis

No.	Elements	Relative Abundance (%)
1	Si	11.850
2	Fe	4.826
3	Al	3.740
4	K	0.487
5	Ca	0.434

Silica Content with Various Concentrations of NaOH and HCl

In silica extraction, various concentrations of NaOH and HCl (2M, 4M, 6M, 8M and 10 M) were used. The yield percent are shown in Table 2 and Figure 3.

Silica content is directly proportion to the concentration of NaOH and HCl. Optimum concentration was found to be 8 M for both NaOH and HCl. Beyond these concentrations the silica content does not change.

Table 2 Yield (%) of Silica with Various Concentrations

No.	Concentration (M)		Weight of silica (g)	Yield of silica (%)
	NaOH	HCl		
1	2	2	1.5	15
2	4	4	1.6	16
3	6	6	2.5	25
4	8	8	3.8	38
5	10	10	3.8	38

Sample - 10 g, Reaction time - 60 min
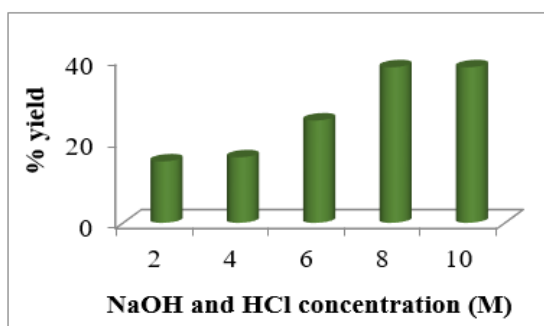
Figure 3 Yield (%) of silica with various concentration of NaOH and HCl

Silica Content with Various Reaction Time of Clay Sample

In silica extraction, various reaction time (60 min, 90 min and 120 min) were used in 8 M NaOH and HCl. The yield percent are shown in Table 3 and Figure 4.

Silica content is directly proportion to the reaction time used. Optimum times was found to be 90 min. Beyond these times, the silica content is decreased.

Table 3 Yield (%) of Silica with Various Reaction Time

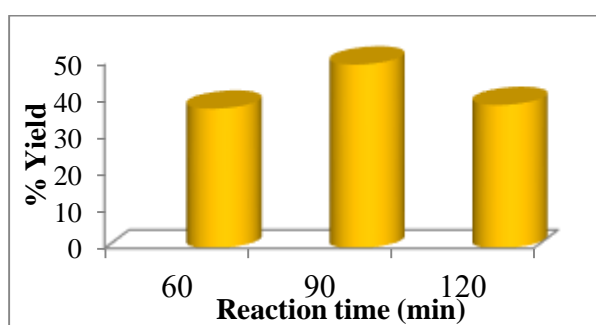| No.	Concentration of NaOH (M)	Concentration of HCl (M)	Time (min)	Weight of silica (g)	Yield of silica (%)
1	8	8	60	3.8	38
2	8	8	90	5.0	50
3	8	8	120	3.9	39

Sample - 10 g

Figure 4 Yield (%) of silica with various reaction time

Silica Content with Various Temperature

In silica extraction, various temperature (60º, 80º and 100º C) were used in 8 M NaOH and HCl. The yield percents are shown in Table 4 and Figure 5.

Silica content is directly proportion to the various temperature. Optimum temperature was found to be 80 ºC. Beyond these temperature, the silica content is decreased.

Table 4 Yield (%) of Silica with Various Temperature

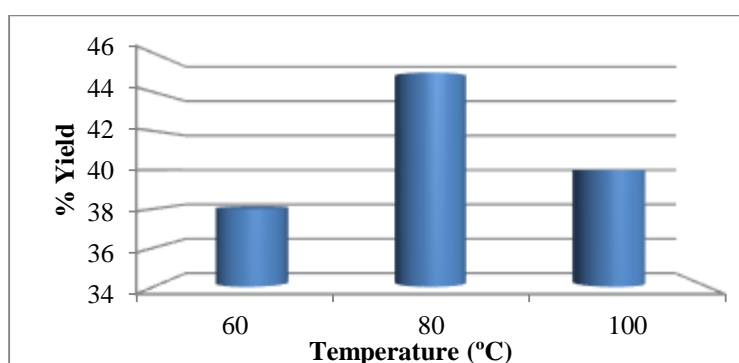| No.	Concentration of NaOH (M)	Concentration of HCl (M)	Temperature (˚C)	Weight of silica (g)	Yield of silica (%)
1	8	8	60	38	38
2	8	8	80	4.5	45
3	8	8	100	4.0	40

Reaction time - 90 min,　　Sample - 10 g

Figure 5 Yield (%) of silica with various temperature

Characterization of Extracted Silica

EDXRF Analysis of Extracted Silica

For qualitative determination of elements in pure silica powder samples, EDXRF technique was used. EDXRF spectrum of the prepared pure silica samples are shown in Figure 6. The relative abundance of metals oxide in these samples are presented in Table 5.

According to EDXRF analysis, it can be observed that silica (SiO_2) contents were found to be the highest (78.162%) in these samples. When the results of EDXRF for pure silica were compared with XRD diffractogram, it was found that these results were agreed with each other.
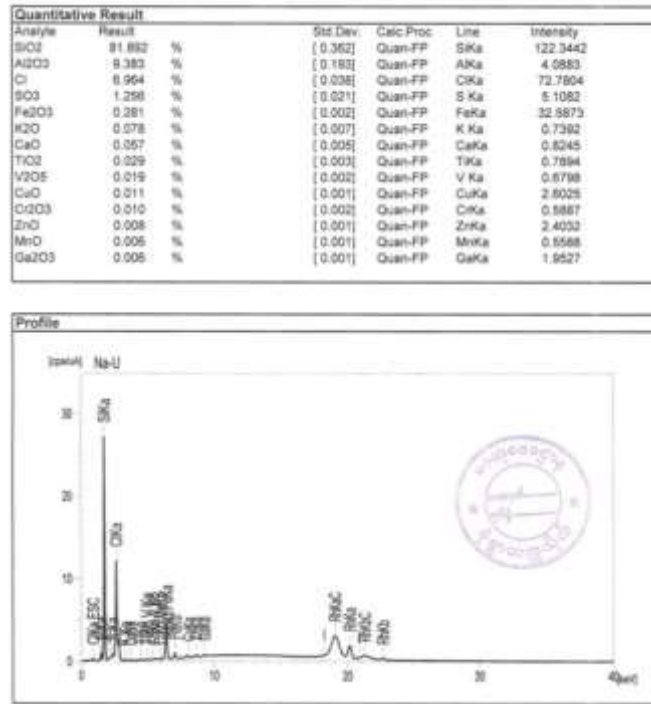
Figure 6 EDXRF spectrum of extracted Silica

Table 5 Relative Abundance of Some Oxides in the Extracted Silica

No	Analyte	Relative Abundance (%)
1	SiO_2	78.162%
2	Al_2O_3	11.519%
3	Cl	8.282%
4	SO_3	1.124%
5	Fe_2O_3	0.635

FT IR Spectra of Pure Silica

The FT IR spectrum of extracted silica sample is shown in Figure 7. The bond assignment of extracted silica sample is tabulated in Table 6. According to FT IR data, the bands at 1052cm^{-1} (Si-O stretching vibration), 789 cm^{-1} (Si-O-Si stretching vibration), 442 cm^{-1} (Si-O in-plane bending vibration) were observed in this sample.
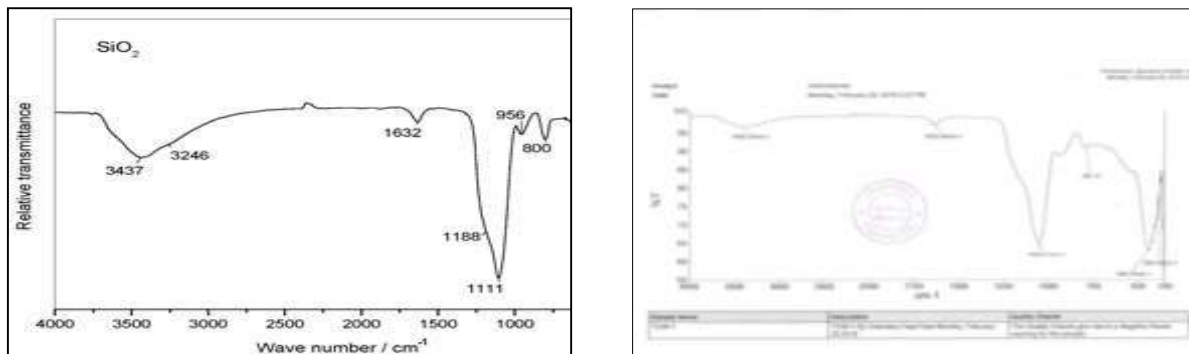

**Figure 7**   FT IR spectra of reference silica and extracted silica

Table 6 Vibrational Mode for SiO₂ Groups per unit Cell of Extracted Silica

Assignment	Spectral region assigned for polymorphs of SiO₂ (cm⁻¹)	Spectral region assigned for polymorphs of SiO₂ (cm⁻¹) Literature value*
Si-O stretching vibration (motion primarily associated with the oxygen atom)	1052	1200 – 1000
Si-O stretching vibration (motion primarily associated with the silicon atom)	789	825 – 600
Si-O bending vibration	442 392	600 – 390
Distortion modes	383	380 – 100

* Ellis, 1958

XRD Diffractogram of Extracted Silica

The X-ray diffraction pattern of extracted silica is shown in Figure 8. These XRD pattern shows ten characteristic peaks (123), (211), (212), (201), (113), (020), (102), (111), (110), (011) for extracted silica. These peaks support the amorphous nature for extracted silica.
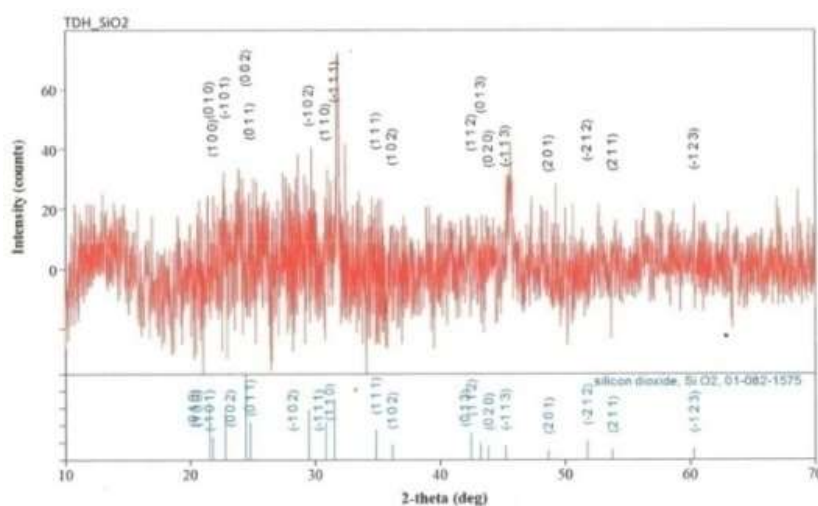
Figure 8 X-ray diffractogram of extracted silica

SEM Analysis of Extracted Silica with 8 M HCl from Clay Powder

SEM measurement for surface analysis was carried on silica from clay mineral. The SEM micrographs of extracted silica are shown in Figures 9 (a) and (b). It can be clearly seen that the crystalline and amorphous nature of silica was observed in SEM images. From surface morphology of silica sample, many small pores and particles with diameter <10 μg were seen on the surfaces of silica sample.

(a) (b)

Figure 9 SEM micrographs of extracted silica from clay powder

Removal Efficiency of Silica

The removal efficiency of silica was determined by using aqueous lead solution (100 ppm) with two methods such as shaking and column methods. Shaking method is more effective than standing method. But shaking method is not benefited for waste water treatment. Thus, only standing method is used for waste water treatment along this research. The percent removal of metal ion (Lead) was described in Table (7).

$$\% \text{ removal of metal ion} = \frac{C_o - C_e}{C_o} \times 100$$

C_o = initial concentration (ppm)

C_e = final concentration (ppm)

Table 7 Percent Removal of Metal Ion (Lead)

Method	Initial concentration (ppm)	Final concentration (ppm)	% removal
Standing	36.51	12.86	64.77

Lead Content in Waste Water

Lead content in waste water determined by using AAS method. This results are described in the following Table 8.

According to AAS results, silica can remove 64.76% of Lead from sample (1) and 34.84% of lead from sample (2). Comparing the removal % of lead it can be clearly seen that the adsorption efficiency is depended on the concentration of adsorbate. These results indicate that the standing method is depended on the flow rate.

Table 8 Determination of Lead Content in Waste Water (Before and After treatment)

No	Lead (ppm)	Before treatment	After treatment	% removal
1	Sample (1)	2.049	0.722	64.76
2	Sample (2)	0.597	0.389	34.84

Characterization of Extracted Silica and Lead Adsorbed Silica

EDXRF spectra of extracted silica

The EDXRF spectrum of extracted silica is shown in Figure 10. These spectra shown 62.891% of silicon and small amount of some minerals are presented in extracted silica.

EDXRF spectrum of lead adsorbed silica (sample-1)

The EDXRF spectrum of lead adsorbed silica for sample (1) is shown in Figure 11. Silica can adsorb 27.194% of lead from waste water.

EDXRF spectrum of lead adsorbed silica (sample -2)

The EDXRF spectrum of lead adsorbed silica for sample (2) is shown in Figure 12. Silica can adsorbed 0.265% of lead from waste water.
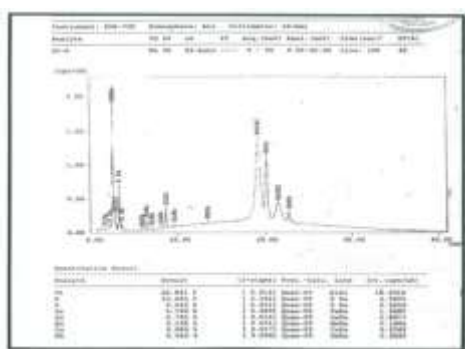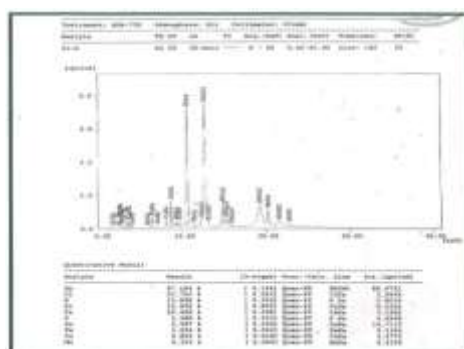

Figure 10 EDXRF spectrum of extracted silica

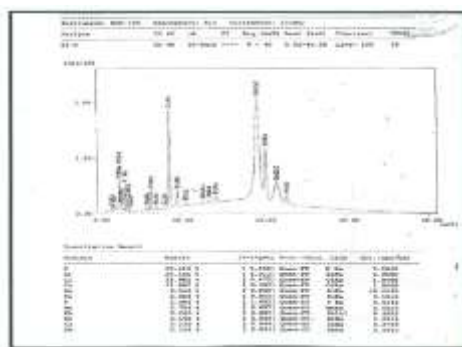**Figure 11** EDXRF spectrum of lead adsorbed silica (sample -1)

Figure 12 EDXRF spectrum of lead adsorbed silica (sample - 2)

Conclusion

In this research work, silica was extracted from Montmorillonite, clay mineral. The clay mineral was collected from Khin Phone Chone Village, Kyaukpadaung Township. The silicon contents found in clay minerals were determined by EDXRF. The observed spectra indicated that the amount of silicon was the highest, 11.850% in clay sample.

In silica extraction, the optimum condition such as NaOH concentration in the range of (2 to 10 M) was determined. 8M NaOH was found to be highest yield 38.00% of silica. Increasing the alkali solution, increase the % yield of SiO_2 till 8.0 M, beyond these concentrations silica content does not significantly change.

The alkaline extraction was investigated at various reaction time of 60, 90 and 120 min. Silica content is directly proportion to the reaction time used. Optimum time was found to be 90 min. Beyond these time, the silica content is decreased. The preparation of silica was investigated at various temperature of 60 ℃, 80 ℃ and 100 ℃. Optimum temperature was found to be 80 ℃.

The extracted silica was characterized by using EDXRF, FT IR, XRD and SEM analysis. From EDXRF data, the highest amount of silica were found to be 81.892% for clay sample.

From FTIR spectra, the peak at 1052 cm^{-1} for extracted silica was indicated the presence of Si-O stretching modes involving motion primarily associated with the oxygen atom. It is obvious that the extracted silica is identical to reference silica.

Crystal structure identification was achieved by X-ray diffraction (XRD), from the XRD diffractogram ten characteristic peaks for SiO_2 (silica) observed in extracted pure silica. SEM image showed that silica from clay has amorphous nature.

The adsorption character of silica was determined by using lead solution   (100 ppm) with two methods, shaking and standing method. It can be absorb (96.65%) for shaking and (64.77%) for standing method, respectively. In this research work, only standing method was used to treatment of wastewater.

In addition, the silica was used in treatment of lead contaminant wastewater from lead-acid battery factory. Silica can reduce pollutant level of wastewater. Toxic heavy metal pollutants cause several environmental problems to environment. The most common heavy metal pollutant is lead. Silica can also reduce lead content in wastewater. The lead content in wastewater sample (1) and (2) were found to be   2.049 ppm and 0.597 ppm respectively.

After treatment, the lead content left was to 0.722 ppm and 0.389 ppm. The % removal was found to be 64.76% for sample (1) and 34.84% for sample (2).

EDXRF spectra data, lead adsorbed silica for sample (1) and sample (2) were found to be 27.194% and 0.265% respectively.

In this research work, one of the abundant clay minerals, montmorillonite can be converted into valuable product (silica) used for several applications in industry. The silica obtained is very amorphous. The pure silica was synthesized from clay sample, successfully.

Acknowledgements

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