

SYNTHESIS AND CHARACTERIZATION OF RICE HUSK SILICA FROM RICE HUSK ASH AND *It's* UTILIZATION FOR POROUS CERAMIC CANDLE FILTER

Naw Mu Htoo¹, Mya Kay Thi Aung², Kyi San Aye³

Abstract

This work concerns with the synthesis and characterization of rice husk silica (RHS) from rice husk ash (RHA) and its utilization for porous ceramic candle filter. Ma Naw Thukha rice husk was used to prepare RHA, by calcinations of rice husk (RH) at 700 °C for 6 h. Preparation of silica powder was carried out from RHA by using precipitation method. The silica from RHA was extracted by 2.4 M NaOH for 3 h and then precipitated by adding concentrated H₂SO₄. The characterization of sample was carried out by modern techniques (SEM, FT IR, XRD, EDXRF) and determination of physicochemical properties using conventional methods. SEM micrograph indicates sponge like nature. XRD diffractogram exhibits an indication of amorphous silica. From FT IR spectral assignment, the band at 1150 to 1000 cm⁻¹ indicates the stretching vibration of Si-O (asym) band in Si-O-Si group. From EDXRF analysis, the highest relative abundance of silica was found to be 99.33 % in RHS. The yield percent of RHS was found to be 52.7 %. Ceramic candle filter (CCF) was prepared by using the raw materials such as RHS, sand, char and clay. The physicochemical properties of CCF were also determined by conventional methods. The performance of ceramic candle filter (CCF) made from a mixture of sand, char, natural clay and RHS in removing chloride from sodium chloride solution was investigated. Removal of chloride ion from sodium chloride solution was studied with different parameters (flow rate for 100 mL fraction: 5, 10, 15, 20, 30, 40, 60 min; different concentrations: 100,200 and 400 ppm). Determination of chloride ion in sodium chloride solution was determined by argentometric method. From the results, the optimum condition for ceramic filter was found to be 69.58 mgg⁻¹ of sorption capacity at a flow rate of 20 min for 100 mL fraction. Removal of chloride ion from lakes of Naung Bo Village (NB) and San Pyaw Village (SP), Naung Bo Group, Ka Wa Township, Bago Region was carried out. From the results, ceramic candle filter (CCF) was **found relativity** as an effective adsorbent for saline water treatment.

Keywords : Rice Husk Ash (RHA), Rice Husk Silica (RHS), calcination, precipitation method, argentometric method, ceramic candle filter (CCF)

¹Lecturer, Department of Chemistry, Military Institute of Nursing and Paramedical Science

² Lecturer, Dr, Department of Chemistry, University of Yangon

³Associate Professor, Dr, Department of Chemistry, Bago University

Introduction

Paddy rice is grown on every continent except Antarctica and the extent of paddy cultivation covers about 1 percent of the earth's surface. More than half of the world's population depends on rice as a staple food and it ranks second to wheat in terms of cultivation area and production. Production of rice is dominated by Asia, where rice is the only food crop that can be grown during the rainy season in the waterlogged tropical areas. Asia generates over 90 percent of world rice production (Noor and Rohasliney, 2012).

Rice husk, as one of the most trifling agricultural products, can act as a good absorbent by absorbing heavy metals and removal of colour. Rice husk ash (RHA) was obtained after burning. The beneficiation of rice husk ash has been used in many applications. RHA can be an economically variable raw material for production of silica gels and powders. The amorphous nature of RHA silica makes it extractable at lower temperature, and hence provides a low energy method. Rice husk ash contains over 80% of silica. (Abedi-Kopai and Mohri-Esfahani, 2012; Rafiee *et al.*, 2012 ; Thudaij and Nuntiya, 2008).

The most important aspect in improving the health of the people is to provide communities with safe and clean water. In this modern age, the 21st century, an estimated 1.1 billion people worldwide still do not have access to safe potable water. A large percentage of these people are from the developing world, especially in the rural areas and low-income communities (WHO, 2007).

Safe drinking water is also an immediate priority in most emergencies. When normal water supplies are interrupted or compromised due to natural disasters, complex emergencies, or outbreaks, responders often encourage affected populations to boil or disinfect their drinking water to ensure its microbiological integrity. Recently, point-of-use water treatment (PoUWT) options verified in the development context have been recommended for use in emergencies (Lantagne and Clasen, 2009).

Point-of-use or household water treatment methods can be used to improve the quality and safety of water for drinking in situations where there is no safe centrally treated water supply or where the treated water supply system has been compromised. Some studies have shown that simple and relatively

inexpensive home water treatment and storage methods can result in substantial improvements in the microbial quality of drinking water and reduced risks of illness and death, even in the absence of improved sanitation. There are four household water treatment systems (HWTS) in removing bacterial and chemical contaminants: the biosand filter (BSF), the bucket filter (BF), the ceramic candle filter (CCF) and the silver-impregnated porous pot filter (SIPP) (Mwabi,2011).

In this study, RHS from RHA will be prepared and characterized by conventional and modern techniques. Ceramic candle filter (CCF) was made with RHS as raw material. Sorption capacity of CCF for the removal of chloride ion from sodium chloride solution were also be investigated.

Materials and Methods

Collection of Rice Husk (RH)Sample

Ma Naw Thukha rice husk was collected from Aung Thuka Rice Mill, Bago Region, Bago Township. The locally available rice husk RH was washed with water and dried at room temperature as shown in Figure 1. The sample was stored in air tight plastic bags.



Figure 1 Rice husk

Collection of Water Samples

Water samples were collected from lakes of Naung Bo and lake of San Pyaw, Naung Bo group, Ka Wa Township, Bago Region. CCF was prepared by using the raw materials such as RHS, sand, char and clay.

Preparation and Characterization of RHS

Preparation of Rice Husk Ash (RHA) from RH

RH (380g) was preheated on electrical hot plate until to obtain char. This rice husk char (RHC) was burnt in porcelain crucible using muffle furnace about 6 h at 700 °C until to obtain a grey ash. After burning, the ash was cooled at room temperature and then passed through the 180 mesh size sieve. The sample was stored in the desiccators at room temperature.

Preparation of Rice Husk Silica (RHS)

10 gram of RHA sample was stirred in 80mL of 2.4 M sodium hydroxide solution. RHA was boiled in a 250 mL beaker with covering at 90-100 °C for 3h. The solution was filtered and the residue was washed with 20mL distilled water. The filtrate was allowed to cool to room temperature and 5 M H₂SO₄ was added until pH 2 and then NH₄OH was added until pH 8 was reached and allowed to cool to room temperature. The filtrate was then dried at 120 °C for 12 h and refluxed with 6 M HCl for 4 h and then washed repeatedly using deionized water to make it acid free. It was dissolved in 2.4 M NaOH solution by continuous stirring for 10 h on a magnetic stirrer and then concentrated H₂SO₄ was added to adjust pH in the range of 7–8. The precipitated silica was washed repeatedly with warm deionized water until the filtrate became completely alkali free and it was dried at 50 °C for 48 h in the oven.

Characterization of prepared samples

Determination of physicochemical properties of prepared sample was done by conventional methods. The results are shown in Table 1. RHS was characterized by modern techniques (SEM, FT IR, EDXRF, XRD).

Preparation of Ceramic Candle Filter and its Sorption Study

Preparation of ceramic candle filter (CCF)

Ceramic Candle Filter (CCF) was prepared by using the raw materials such as RHS, sand, char and clay. The process for making of ceramic candle

were grinding, screening, mixing, modeling drying and firing. The filter was equipped with a flow rate controller as shown in Figure 2 (a) and (b).

Determination of physicochemical properties of CCF

Determination of physicochemical properties of CCF was carried out using conventional methods. The results are shown in Table 3.

Determination of sorption study of prepared CCF

Removal of chloride ion from sodium chloride solution was studied with different parameters (flow rate for 100 mL fraction : 5, 10, 15, 20, 30, 40, 60 min, different concentrations : 100, 200, 400 ppm). Concentration of chloride ion in sodium chloride solution was determined by Argentometric method. The results were shown in Tables 4,5 and Figures 7 (a, b, c, d, e, f, g), Figure 8.

Removal of Chloride Ion from Lakes of Naung Bo and San Pyaw by CCF

The saline water collected from lakes of Naung Bo and San Pyaw were treated for the removal of chloride ion by CCF at a flow rate of 20 min for 100 mL fraction. The results are shown in Table 6 and Figure 9.



(a)



(b)

Figure 2. (a) Ceramic
(b) Ceramic Candle Filter

Results and Discussion

Physicochemical Analysis of Prepared Samples

Rice husk ash (RHA) was obtained by heating rice husk in muffle furnace for 6 h at 700°C. RHS was prepared from RHA using precipitation method.

The characteristic of the physicochemical properties of prepared samples were determined. Table 1 shows the physicochemical properties of prepared samples. It was found that pH value is nearly neutral. Bulk density and moisture content of RHA were smaller than that of RHS.

Table 1 Physicochemical Properties of Prepared Samples

Sample	pH	Moisture content (%)	Bulk density (g mL ⁻¹)
RHA	6.5	4	0.29
RHS	7.5	8	0.45

Vogel, 1968

Characterization of RHS

SEM analysis

Surface morphology of sample was observed by SEM analysis. SEM micrograph of RHS is shown in Figure 3. It indicated the sponge like nature of RHS.

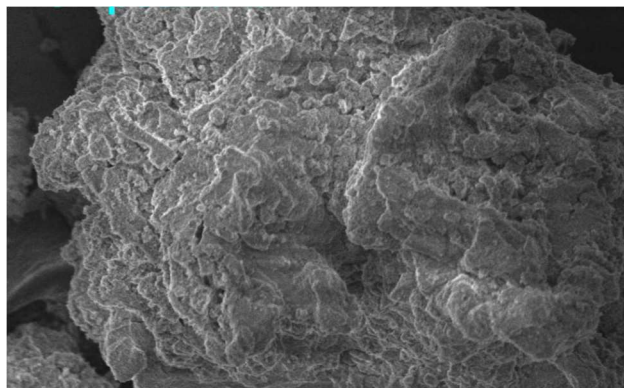


Figure 3 SEM microphotograph of RHS

FT IR analysis

FT IR analysis of silica is presented in Figure 4. The result is shown in Table 2. From the FT IR spectral assignment, the strong bands at a range from 1150 cm^{-1} to 1000 cm^{-1} are due to asymmetric stretching mode of Si-O-Si group.

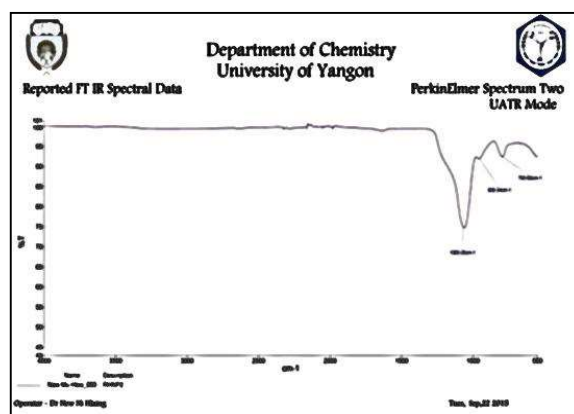


Figure 4 FT IR spectrum of RHS

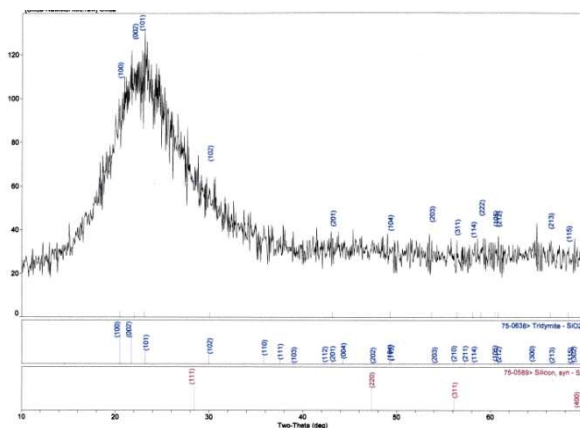
Table 2 FT IR Spectral Assignment of RHS

Observed Frequency (cm^{-1})	*Literature Frequency (cm^{-1})	Band Assignment
1060	1150-1000	$\nu_{\text{Si-O(asy)}}$

*Nakamoto, 1986

XRD analysis

The X-ray diffraction pattern of RHS is shown in Figure 5. The XRD pattern exhibits broad maximum, extending 2θ value from 19° to 25° for RHS which is an indication of amorphous silica.

**Figure 5** XRD spectrum of RHS**EDXRF analysis of prepared sample**

The EDXRF spectra exhibited the relative abundance of silica in RHS described in Figure 4. From these results, it was obvious that the relative abundance of SiO₂ was high in RHS. Other compounds (K₂O, CaO, Fe₂O₃, MnO, ZnO, and CuO, etc) were also observed. The relative abundance of silica was 99.33 %.

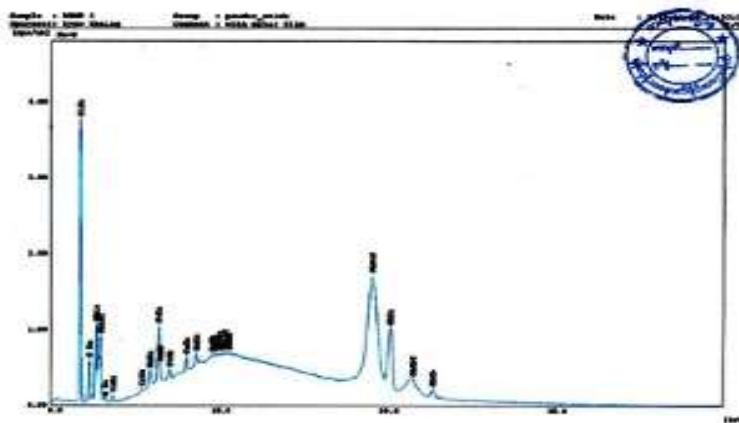


Figure 6 EDXRF spectrum of RHS

Characterization of Ceramic Candle filter (CCF)

Determination of Physicochemical Properties of CCF

The results of bulk density, porosity, water absorption and flow rate are shown in Table 3.

Table 3 Physicochemical Properties of CCF

Bulk Density (g mL⁻¹)	Porosity (%)	Water Absorption (%)	Flow rate (L/h)
0.71	53.39	67.63	1.4

ASTM (2010)

Removal of chloride ion from sodium chloride solution by CCF with different flow rates

Removal of chloride ion from sodium chloride solution with different flow rates for 100 ml fraction in 5,10,15,20,30,40,60 min can be seen in Figure 7 (a, b, c, d, e, f, g). The results are shown in Table 4. It can be observed that sorption capacity increased as the flow time increased but decreased after 20 min flow time was used.

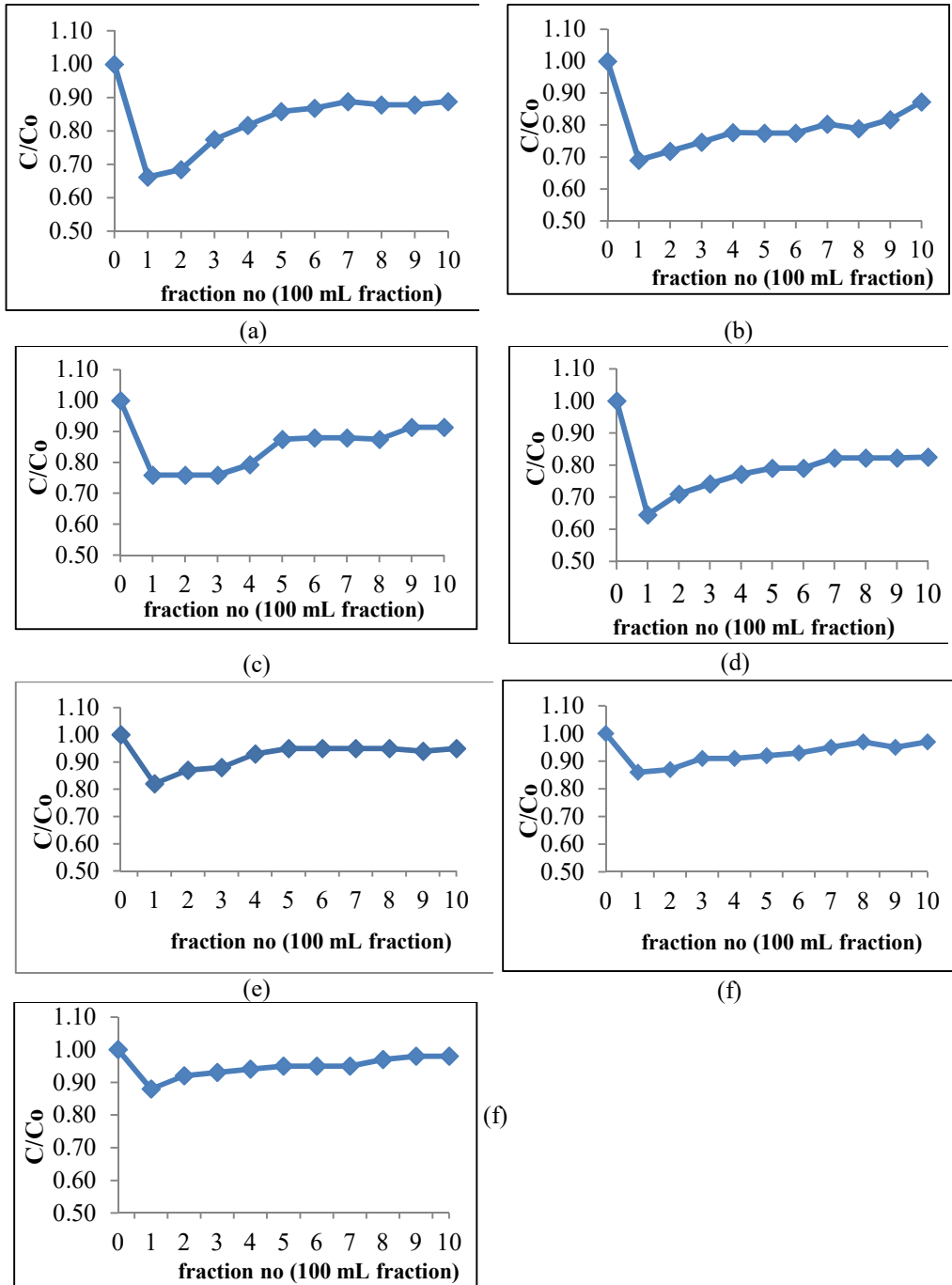


Figure 7 : Breakthrough curve for removal of chloride ion from 100 ppm NaCl solution by CCF with a flow rate of 100 mL fraction for (a) 5 min (b) 10 min (c) 15 min (d) 20 min (e) 30 min (f) 40 min (g) 60 min

Table 4 Removal of Chloride Ion with Different Flow Rates

Flow time for 100 ml fraction(min)	Sorption capacity of CCF (mg g ⁻¹)
5	29.82
10	39.96
15	49.70
20	69.58
30	49.70
40	29.82
60	19.88

Removal of chloride ion from sodium chloride solution by CCF with different concentrations

The effect of initial concentration of sodium chloride was studied using various concentrations such as 100, 200, 400 ppm. The removal percent of chloride ion by CCF decreased as the initial concentration was increased. The removal percent of chloride from 100 ppm sodium chloride solution was found to be the highest value (36%). The results are shown in Table 5 and Figure 8.

Table 5. Percent Removal of Chloride Ion from Sodium Chloride Solution by CCF

Initial concentration (ppm)	Final concentration of chloride ion (ppm)	Percent removal(%)
100	64	36
200	148	26
400	336	16

Flow rate for 100 ml fraction = 20 min

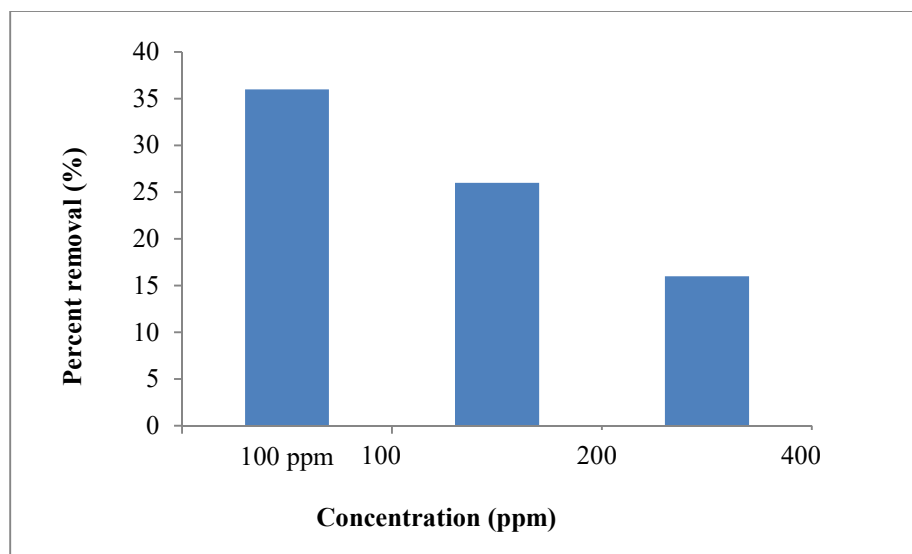


Figure 8 Percent removal of chloride ion in 100 ppm, 200 ppm and 400 ppm by CCF with a flow rate of 100 mL fraction for 20 min

Removal of Chloride Ion from Lakes of Naung Bo and San Pyaw by CCF

The saline water collected from lakes of Naung Bo and San Pyaw were treated for the removal of chloride ion by CCF. The results are shown in Table 6 and Figure 9. CCF is the effective and efficient adsorbent for removal of chloride ion in SP (2) compared to NB and SP (1).

Table 6 Percent Removal of Chloride ion by CCF

Sample	Initial concentration of chloride ion (ppm)	Final concentration of chloride ion (ppm)	Percent removal (%)
NB	55	38	31
SP (1)	527	476	10
SP (2)	69	46	33

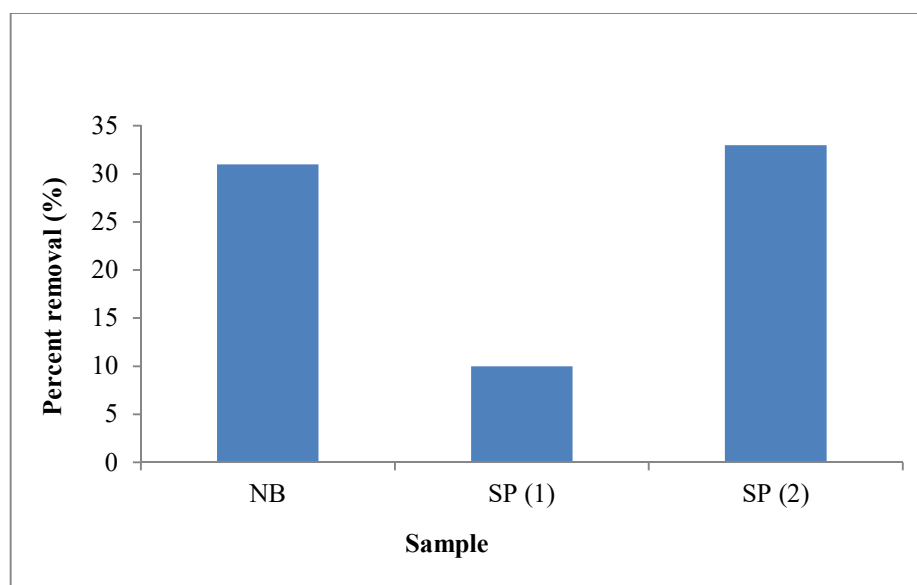


Figure 9 Percent removal of chloride ion in N B, SP (1) and SP (2) by CCF with a flow rate of 100 mL fraction for 20 min

Conclusion

Ma Naw Thu Kha rice husk was collected from Aung Thukha Rice Mill, Bago Township, Bago Region. Rice Husk Ash (RHA) was prepared from Rice Husk (RH) by calcinations at 700°C for 6 h in muffle furnace. Preparation of rice husk silica (RHS) was carried out from RHA using precipitation method. From the results of physicochemical properties determination, the pH of the samples were nearly 7. The bulk densities of RHA and RHS were 0.29 and 0.45 g mL⁻¹ respectively. The moisture content of RHA and RHS were found to be 4 % and 8 % respectively. From SEM analysis, RHS was found to have sponge like nature. From FT IR spectral assignment, the frequency between 1150 to 1000 cm⁻¹ indicates the stretching vibration of Si-O (asym) band indicating that silica is present in RHS. According to XRD diffractogram, RHS exhibited broad maximum extending 2θ values from 19° to 25°. An indication of amorphous silica was observed. From ED XRF spectrum, the

highest relative abundance of silica was found to be 99.33% in RHS. CCF was prepared by using the raw materials such as RHS, sand, char and clay. The bulk density of CCF was found to be 0.71 g mL^{-1} . The porosity, flow rate and water absorption of CCF was found to be 59.39 %, 1.4 L h^{-1} and 67.63 % respectively. The sorption capacity of CCF was observed the highest in the removal of chloride from 100 ppm sodium chloride solution with the flow rate of 20 min for 100 mL fraction. Moreover, CCF was studied for the removal of chloride ion from lakes of Naung Bo and San Pyaw. CCF is the effective and efficient adsorbent for removal of chloride ion in SP (2) compared to NB and SP (1).

Acknowledgements

The authors would like to express their profound gratitude to the Department of Higher Education, Ministry of Education, Myanmar, for giving opportunity to do this research and Myanmar Academy of Arts and Science for allowing to present this paper.

References

- Abedi-Koupai, J. and Mohri-Esfahani, E. (2012). "Desalination of Water Using Nanoparticles of Husk Ashes in Sand Filter". *Proceedings of the 4th International Conference on Nanostructures (ICNS4)*, pp. 1150-1152
- ASTM. (2010). *Standard Test Method for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water*. United States: ASTM International, pp. 6-8
- Lantagne, D. S. and Clasen, T. (2009). "Point of Use Water Treatment in Emergency Response". London: *London School of Hygiene and Tropical Medicine*, vol. 80(5), pp. 819-823
- Mwabi, J. K., Adeyemoand, F. E. and Mahlangu, T. O. (2011). "Household Water Treatment Systems: A Solution to the Production of Safe Drinking Water by the Low-income Communities of Southern Africa". *Physics and Chemistry of the Earth*, vol. 36, pp. 1120-1128
- Nakamoto, K. (1986). *Infrared and Raman Spectra of Inorganic and Coordination Compounds*. New York: 2nd Edition, John Wiley and Sons, Inc., pp. 200-201
- Noor, S. S. and Rohasliney, H. (2012). "Rice Husk as Biosorbent". *Health and the Environmental Journal*, vol. 3(1), pp. 89-95.
- Rafiee, E., Shahebrahimi, S., Feyzi, M. and Shaterzadeh, M. (2012). "Optimization of Synthesis and Characterization of Nano Silica Produced from Rice Husk (Common Waste Material)". *International Nano Letters*, vol. 2(29), pp. 1-8
- Thudajj, N. and Nuntiya, A. (2008). "Preparation of Nanosilica Powder from Rice Husk by Precipitation". *Chiang Mai Journal Science*, vol. 35(1), pp. 266-211
- Vogel, A.I. (1968). *Quantitative Inorganic Analysis*. London: 3rdEd., Longmans Co. Ltd., pp. 116-120
- World Health Organization.(2007). *Desalination for Safe Water Supply*. Geneva: pp. 35-36

