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Preparation and characterization of activated carbon from coconut shell

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Abstract

Fourteen samples of activated carbon were prepared by carbonization and chemical activation of coconut shell. Carbonization was achieved at 500 ºC for 2 hours while chemical activation was achieved by first impregnating the prepared raw coconut shell charcoal with activating reagents, and then activating at 900 ºC. Characteristics of the activated carbons were determined using standard methods. The best activating agent is sulphuric acid and the optimum concentration of activating agent is 10%. The soaking time was 48 hours and the activation time was 4hours to get maximum iodine number. Removal of pollutants from river water and ground water were satisfactory for filter with prepared activated carbon, gravel and silica sand. Results reveal that quality activated carbon could be produced from agro waste coconut shell.

Keywords: activated carbon, coconut shell, waste water, filter

1. INTRODUCTION

Water is essential need for every person in the world for long life. Water covers 70% of the earth's surface and makes up over 60% of the human body. Modern technology demand more and more water, the ways must be constantly devised to tap new resources and to make reuse of water. There are many types of treatment that can improve water quality [1]. Efficient techniques for the removal of highly toxic organic compounds from water have drawn significant interest. A number of methods such as coagulation, filtration with coagulation, precipitation, ozonisation, adsorption, ion exchange, reverse osmosis and advanced oxidation processes have been used for the removal of organic pollutants from polluted water and waste water. These methods have

been found to be limited, since they often involve high capital and operational costs. On the other hand, ion exchange and reverse osmosis are more attractive processes because the pollutant values can be recovered along with their removal from the effluents. Reverse osmosis, ion exchange and advanced oxidation processes do not seem to be economically feasible because of their relatively high investment and operational cost. Among the possible techniques for water treatments, the adsorption process by solid adsorbents shows potential as one of the most efficient methods for the treatments and removal of organic contaminants in waste water treatment. Adsorption has advantages over the other methods because of simple design and can involve investment in term of both initial cost and land required. The adsorption process is widely used for treatment of industrial waste water from organic and inorganic pollutants and meets the great attention from the researchers. In the recent years, the search for low- cost adsorbents that have pollutant – binding capacities has intensified. Materials locally available such as natural materials, agricultural wastes and industrial wastes can be utilized as low- cost adsorbents [2].In environment field activated carbon adsorption has numerous applications in removing pollutants from air or water streams both in the field and in industrial processes such as spill cleanup, ground water remediation, drinking water filtration, air purification, volatile organic compounds capture from panting, dry cleaning, gasoline dispensing operations, and other processes. In medical applications activated carbon is used to treat poisonings and overdoses following oralingestion. It is thought to bind to poison and prevent its absorption by the gastrointestinal tract. In cases of suspected poisoning, medical personal administer activated charcoal on the scene or at a hospital's emergency department [3]. Activated carbons are increasingly used as the economic and stable mass separation agent for the removal of surfactants to raise the final product quality many industrial processes. Activated carbons also play an important role in many areas of modern science and technology such as purification of liquids and gases, separation of mixtures and catalysis. Adsorption of activated carbon is governed by the chemical nature of the aqueous phase, the solid phase and the chemical nature of the absorbing organic .The purpose to carry this research is to investigate the quality of activated carbon from coconut shell by chemical activation using chemicals such as zinc chloride, sulphuric acid and phosphoric acid for different concentration. To identify the effectiveness of the activated carbon, the sample waters are being check before and after using the following tests, pH determination, metal contents by atomic adsorption spectroscopy and total dissolved solid test using TDS meter. After analyzing the samples with different parameters it showed the improvement of pH by decreasing the pH value. From the removal of total suspended solids, total dissolved solids and some metals, it has a lot of efficiency in water treatment.

2 MATERIALS AND METHODS

2.1 Required Materials

The Coconut shell, wood fuel, phosphoric acid, sulphuric acid, zinc chloride, hydrochloric acid, distilled water, clay etc., are required for this research.

2.2 Required Instruments

The Iron furnace(wood fuel furnace), muffle furnace (electric furnace), glass oven, vacuum drying oven, digital rough balance, analytical balance, crucible (graphite), IR thermometer (laser gun), grinder, ball mill, motor and pestle, sieves, nitrate testing kit, phosphate testing kit, beaker, conical flask, pH meter etc., are needed for this research.

2.3 Collection of Raw materials

Coconut trees have a smooth, columnar, light grey-brown trunk, with a mean diameter of 30-40cm height,

and topped with a terminal crown of leaves. Tall selections may attain a height of 24-30m. Shell charcoal is used widely as domestic and industrial fuel. It is also used by black smith and gold smiths and in laundries. Shell charcoal is also used to produce activated carbon. Activated carbon produced from coconut shell has certain specific advantages as the raw material can adsorb certain molecular species [4]. The coconut shells which are raw materials used for the production of activated carbon were collected and then pre-treated before activated carbon was produced.

2.4 Pre-treatment of raw material

The pith and coir were removed from raw coconut shell to reduce the tar in the formation of charcoal.

2.5 Production of charcoal from coconut shell

The pre-treatment coconut shell was placed in iron container with lid and carbonized in traditional furnace using wood as fuel at 500 °C for 2 hours. The temperature of furnace was detected by infrared thermometer. The obtained coconut shell charcoal was grinded with ball maill and then sieved with mesh size No.10 and mesh size No.20 to obtain size of particles between 2mm and 0.842mm and less then 0.842mm. Then charcoals with different particle size were collected for activation process.

2.5.1 Process selected

Activated carbon is a form of carbon species that is processed and prepared to average high porosity and very large surface area available for adsorption. The large surface area implies a high capacity for adsorbing chemicals from gases and liquids. There are two types of activation of carbon, chemical and physical activations.

Chemical activation:

Chemical activation is commonly used because it is easier and required low temperature and less time. Chemical activation is done by activating chemical solution can be strong acid or strong base or salt for example(sodium chloride, zinc chloride and calcium chloride). The carbon structure is filled with pores which is closed, so by this chemical solution, the pores will opened and then impurities will struck in that pores of activated carbon[5].

2.5.2 Chemical activation procedures

2.5.2.1 Preparation the soaking solutions

Zinc chloride, sulphuric acid and phosphoric acid were selected as activating agents and prepared the two concentrations for one activating agent to know the effectiveness of the concentration on activation of the carbon. For zinc chloride solution, 10% hydrochloric acid solution was used as solvent and prepared the different concentrations (10% and 25%). The 10% and 20% solutions were made for sulphuric acid and phosphoric acid.

2.5.2.2 Chemical activation with chloride salt, H_2SO_4 solution and H_3PO_4 solution

The material after carbonization is impregnated with both of the two concentrations of chloride salt solutions (zinc chloride), sulphuric acid solutions and phosphoric acid solutions for 48 hours, 96 hours and 168 hours. 1:2w/v of material to soaking solution is used so that the material get well adsorbed the solution

for a period of time. At the end of the soaking time, the excess chloride solution was decanted off and air dried. Then the materials were placed in graphite crucible with lid and closed the liner of the lid with clay to create anaerobic condition. After that it was carbonized again in muffle furnace at 900 ºC in the heating rate of 5 ºC per minute for a period of 2hours and 4 hours. After the carbonization, the carbon was washed sufficiently with 10% HCl in hot plate at 110 ºC for 20 minutes to remove the cations. Then the materials were washed with plenty of boiling water to remove the excess acid until getting pH-7 and then dried in furnace at 300 ºC for 2 hours or dried in the oven at 150 ºC for 4 hours. After that, all the samples were stored in closed type bottles for applications.

2.6 Characterization of the carbons

Moisture content (%) by mass, ash (on dry basis) % by mass, volatile matter, fixed carbon content, bulk density, iodine number were analyzed as per the standard procedures. pH was analyzed by using EUTECH pH meter (model pH700). % yield of the activated carbons was calculated.

2.7 Collection of water samples

The polluted water samples were collected from Yangon river, near Kyeemyindine township, Hlaing river near Aung Zay Ya Bridge and tap water from department of research and innovation (DRI) for the study purpose.

2.8 Pretreatment of the adsorbents

Gravel, silica sand and the activated carbon were used as adsorbents for the polluted water treatment. Before using as absorbents in the filter, all the adsorbents were upgraded by washing with purified water to remove the dusts and impurities from gravel and silica sand. The activated carbon (iodine number=519.7mg/g) with particle size between 2mm and 0.842mm was chosen for filter. Most of the size of activated carbon used as an adsorbent for water purification in filter cartridge is 8 mesh size of activated carbon, so some of the small particles were removed by washing with water.

2.9 Preparing a filter for treatment of water samples

A filter for the treatment of polluted waters was built by using the plastic boxes. The bottom of plastic box was made a hole and put 200g of each for gravel, silica sand and activated carbon with maximum iodine number in each plastic box. The plastic boxes were fitted on each other by the following arrangements. The bottom plastic box contains activated carbon, the second box contains silica sand and the top plastic box contains gravel.

2.10 Treatment of water samples

Three liters of the different water samples were flowed in the filter by flow rate with 66.66ml/min and collected with a clean plastic bucket. The water quality parameters were checked for physico-chemical parameters as per standard methods before and after treatment and the efficiency dose of activated carbon was determined.

3 RESULTS AND DISCUSSION

Kimet al; 2001 revealed that activated carbon produced from coconut shells typically have a tighter, more micro porous pore structure than their coal based counter parts. They confirmed that this is due to the inherent

pore structure of the raw material coconut shell as compared to raw material coals. This micro porosity lends itself towards certain applications where activated carbon is used.

Table1. Activation conditions of coconut shell charcoal

Type of Sample	Size of Sample	Activating agent	Soaking time (days)	Activating temperature (°C)	Activating time (hours)	Drying temperature (°C)	Drying time (hours)
AC-1	<2mm and >0.842mm	10% $ZnCl_2$	7	900	2	300	2
AC-2	□	25% $ZnCl_2$	7	900	2	300	2
AC-3	□	10% H_2SO_4	7	900	2	300	2
AC-4	□	20% H_2SO_4	7	900	2	300	2
AC-5	□	10% H_2SO_4	4	900	4	150	4
AC-6	< 2 mm and >0.841mm	10%H_3PO_4	4	900	4	150	4
AC-7	□	10%H_2SO_4	2	900	4	150	4
AC-8	□	10%H_3PO_4	2	900	4	150	4
AC-9	< 0.841mm	25%$ZnCl_2$	7	900	4	150	4
AC-10	□	10%H_2SO_4	7	900	4	150	4
AC-11	□	10%H_2SO_4	4	900	4	150	4
AC-12	□	10%H_3PO_4	4	900	4	150	4
AC-13	□	10%H_2SO_4	2	900	4	150	4
AC-14	□	10%H_3PO_4	2	900	4	150	4

Table-1shows the list of activated carbons (ACs) prepared from coconut shell and method of activation of each AC1 to AC14 were prepared by chemical activation method. The temperature of carbonization and activation were carefully selected to maximize development of carbon molecular architecture. Char formation

was made at 500 ˚C for 2 hours. It was observed that charring occurred only in cases where the atmosphere was partially inert. In search of alternative sources and cheaper carbons, agro wastes with average carbon content of 35 per cents have attracted the researchers [7]. In this research, fourteen samples (activated carbons) were prepared with different activating agents, different soaking time, and different activating temperature with different activating time. After that, their characterizations were investigated by standard methods. Moisture content was measured according to (ASTM 2867-99), ash content by ASTM D2866 and the iodine number by ASTM D4607.

Table-2 physico - chemical properties of prepared activated carbons

Type of sample	Type of Properties						Yield(%)
	Moisture Content (%)	Ash Content (%)	Volatile matter (%)	Fixed Carbon (%)	Iodine number (mg /g)	Density (g/cm3)	
C	13.27	0.56	82.09	4.08	-	-	24.206
AC- 1	10.33	10.63	13.29	65.75	31.16	0.58	14.39
AC- 2	4.87	2.87	10.54	81.72	33.7	0.653	12.96
AC- 3	5.7	2.44	11.81	80.05	61.7	0.646	13.49
AC- 4	3.92	1.94	12.31	81.83	56.71	0.653	13.85
AC- 5	2.0	0.49	11.06	86.45	126.88	0.691	13.6
AC- 6	1.88	0.32	9.63	88.17	176.5	0.712	13.99
AC- 7	1.92	0.43	9.55	88.1	519.7	0.669	14.12
AC- 8	2.05	3.13	9.35	85.47	421.65	0.683	13.85
AC- 9	5.40	0.61	9.41	84.58	271.57	0.920	13.36
AC-10	4.83	0.73	9.62	84.82	411.73	0.847	13.45
AC-11	2.51	0.41	8.03	88.99	454.38	0.695	13.08
AC-12	4.53	2.81	10.03	82.63	312.31	0.888	14.06
AC-13	2.02	0.47	11.06	86.45	570.60	0.609	13.55
AC-14	6.98	2.77	13.13	77.12	383.67	0.887	12.96

Table-2 shows the prepared activated carbons with their physico - chemical properties.

The carbonization of the precursor, the coconut shells led to a char with solid yield of approximate 24%. It was revealed that activated carbon with 519.7mg/g of iodine number is particle size between 2mm and 0.842mm and the iodine number of activated carbon with particle size less than 0.842mm is 570.60.It could due to the finer the particle size, the better the access to the surface area and the faster the rate of soaking the activating agent on the carbon in the same activating condition with the same activating agent. According to the bulk density and iodine number of activated carbons, the optimum conditions to get maximum iodine number with 570.6 mg/g are particle size less than 0.842mm of activated carbon using sulphuric acid as activating agent with 10% concentration, 48 hours for soaking time, 900 ˚C for activating temperature, 4 hours for activating time. Yield % of all the samples are nearly the same. AC 1, 2, 3 and 4 have lowest iodine number with smaller value of bulk density but the value of moisture contents are larger than other samples .According to theory of porosity, the smaller the value of bulk density.

Table3. shows the physico-chemical parameters in both river water (surface water) and ground water as per standard methods before and after treatment with activated carbon obtained maximum iodine number.

Table3- the physico-chemical parameters in both river water (surface water) and ground water as per standard methods before and after treatment with filter

No	Measurable Parameters	Yang-on river water (BT)	Yang-on river water (AT)	Hlaing river water (BT)	Hlaing river water (AT)	Tap water (BT)	Tap water (AT)	WHO guideline standard	Standard value of MNDWQS
1.	Arsenic(mg/L)	0.005	<0.005	0.005	ND	ND	ND	0.05	0.05
2.	Lead(mg/L)	ND	ND	ND	ND	ND	ND	0.01	0.01
3.	Manganese (mg/L)	0.210	0.012	0.267	0.005	0.225	0.035	0.5	0.4
4.	Total Hardness (mg/L)	155	76	88	151	40	35	500	500
5.	Iron(mg/L)	7.784	0.015	9.556	ND	0.143	ND	0.3	1
6.	pH	7.25	7.08	7.92	7.10	7.12	7.06	6.5-8.5	6.5-8.5
7.	Total Dissolved Solid(mg/L)	726	584	349	239	51.7	37	1000	1000
8.	Calcium(mg/L)	30.5	24.19	21.75	17.05	6.3	8.97	200	200
9.	Magnesium(mg/L)	2.804	2.747	2.663	2.693	2.386	2.500	150	150
10.	Nitrate-nitrogen (mg/L)	94.38	2.619	185.38	5.4645	21.045	1.287	50	50
11.	Total alkalinity (mg/L)	240	212	180	102	80	41	3	3
12.	Zinc(mg/L)	0.001	ND	0.004	ND	ND	0.0014	0.5	0-0.5
13.	Total Suspended solid (mg/L)	144	Nil	196	Nil	31	Nil	250	250
14.	Phosphorus(mg/L)	25.33	2,633	28.33	5.466	4.83	0.4	-	-

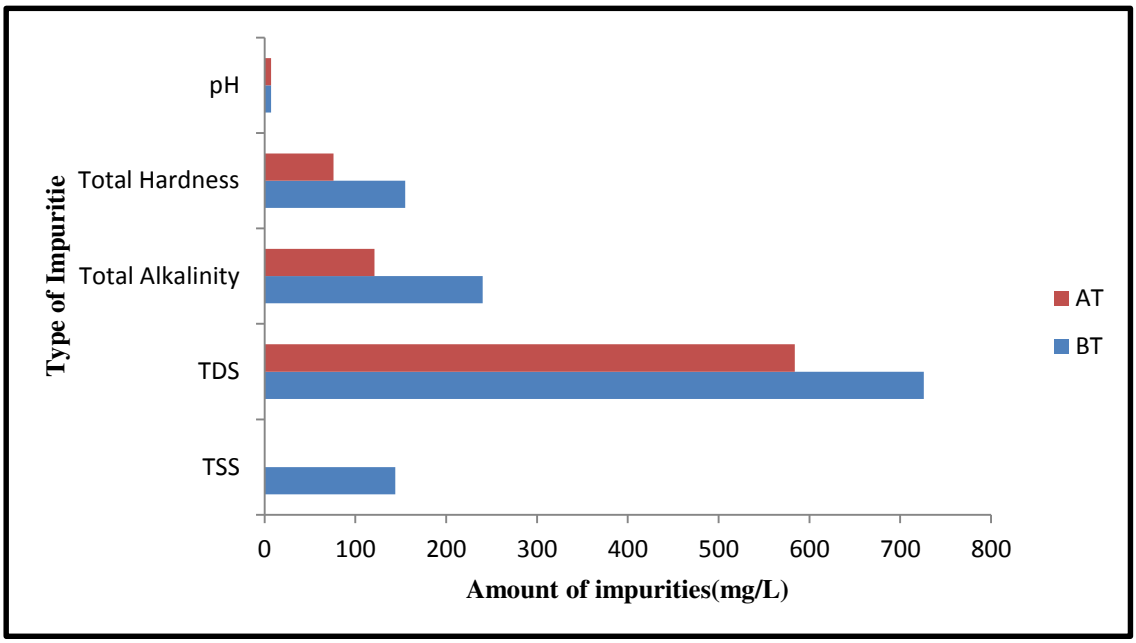

Fig-1.Physico-chemical properties of Yangon river water before and after treatment

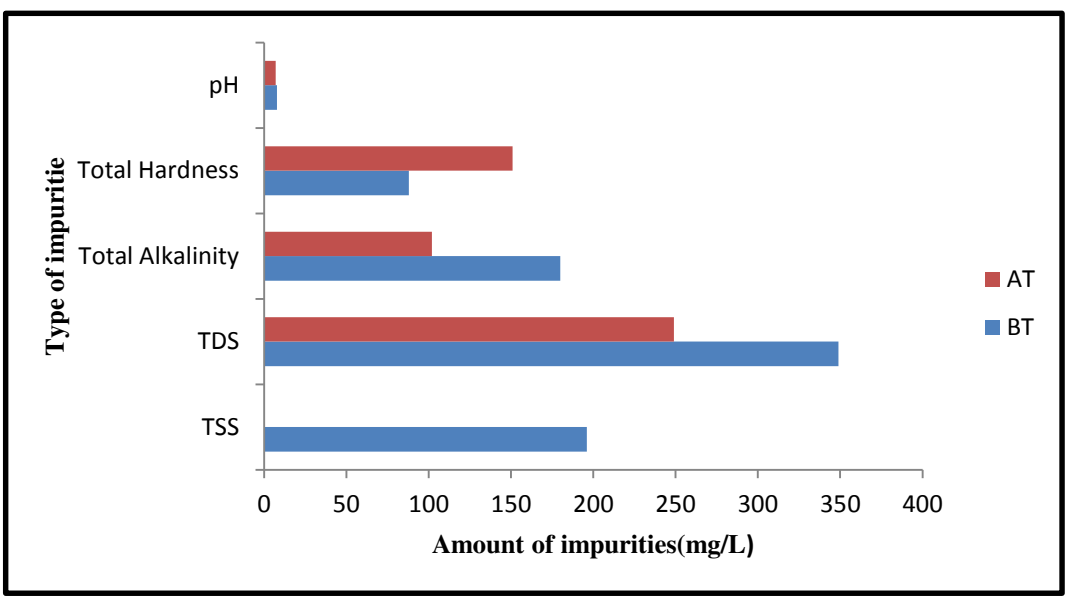

Fig-2.Physico-chemical properties of Hlaing river water before and after treatment

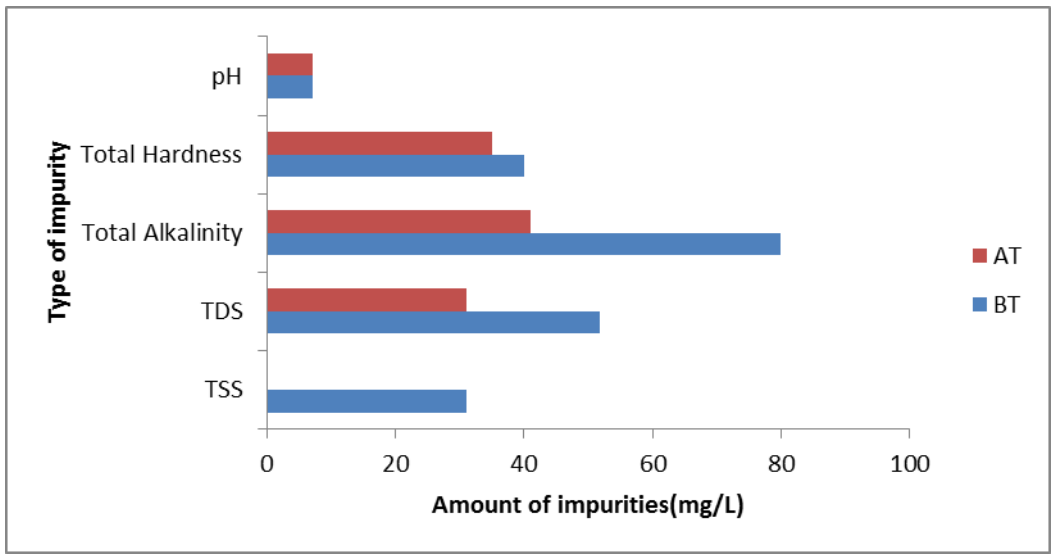

Fig-3.Physico-chemical properties of tap water before and after treatment

The physico – chemical parameters in three water samples as per standard methods before and after treatment. The pH of Yangon river water was 7.25, TDS was 726 mg/L, TSS level was 144mg/L, total alkalinity was 240 mg/L and total hardness was 155mg/L. The physico - chemical parameters of Hlaing river water and tap water were as followed: pH was 7.92 in Hlaing river water and 7.12 in tap water, TDS was 349 mg/L in Hlaing river water and 51.7 in tap water, total alkalinity was 180mg/L in Hlaing river water and 80mg/L in tap water, total hardness was 88mg/L in Hlaing river water and 40mg/L in tap water. The comparative values of all three water samples before and after treatment with filter were shown in figure1, figure2 and figure3. According to the parameter values of three types of water samples after treatment, TSS value could be reduced to zero in all types of samples by filter. TDS was decreased to 584mg/L in Yangon river water, 239mg/L in Hlaing river water and 37mg/L in tap water.30% of TDS could be removed from all three samples. It was found that the filter could remove 50% of total alkalinity from all three samples although the filter could remove 30 % of total hardness from Yangon river water. Total hardness values of Hlaing river water sample was moderately increased after treatment. In tap water, total hardness was reduced from 40mg/L to 35mg/L. pH value of all three samples was reduced a little after treatment.

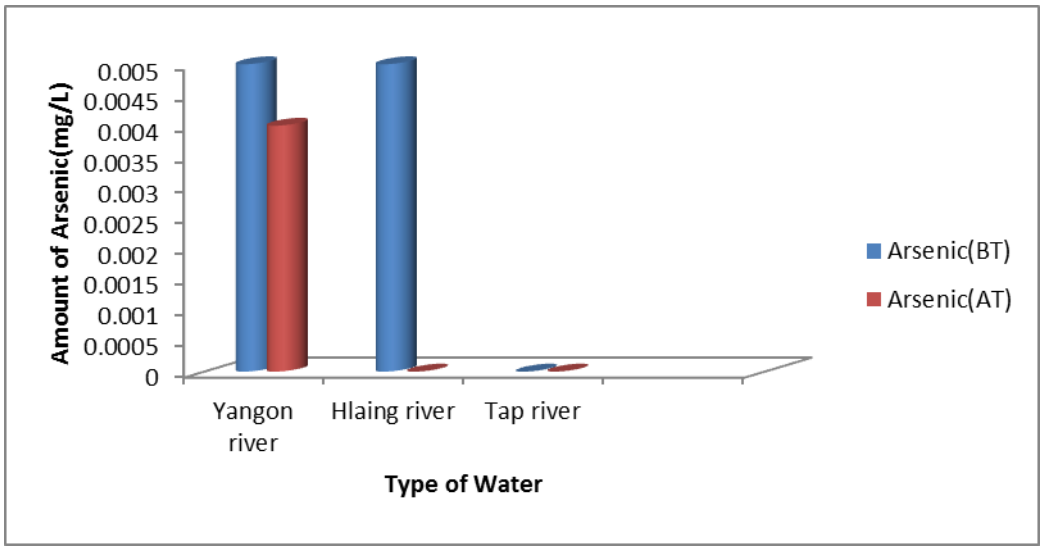

Figure 4- Removal of arsenic from water samples

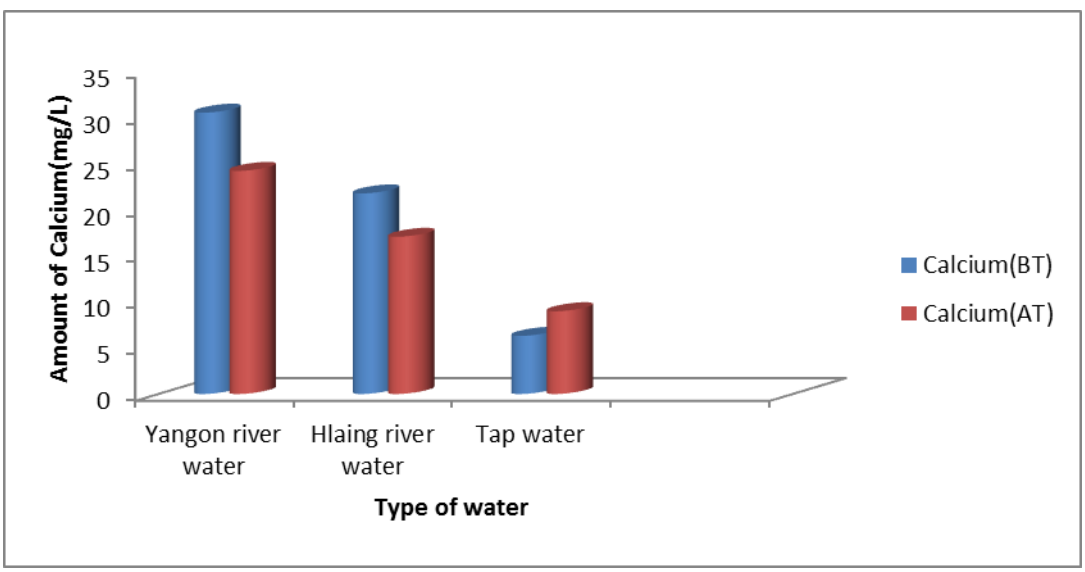

Figure 5- Removal of calcium from water samples

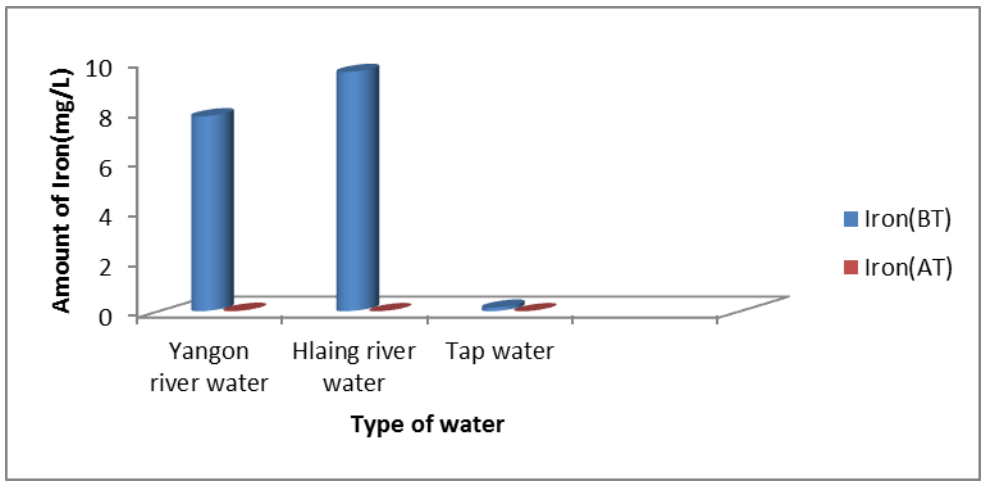

Figure 6- Removal of iron from water samples

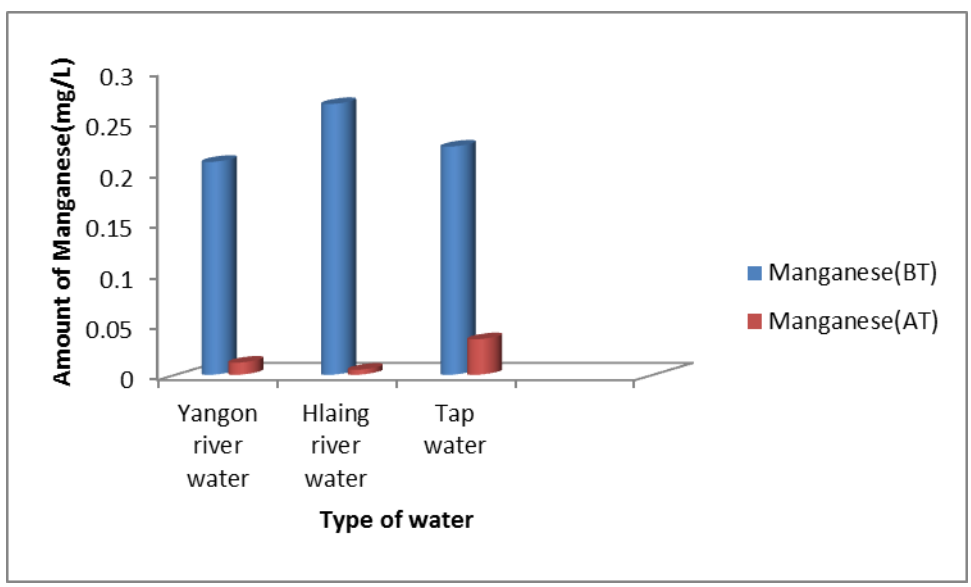

Figure 7- Removal of manganese from water samples

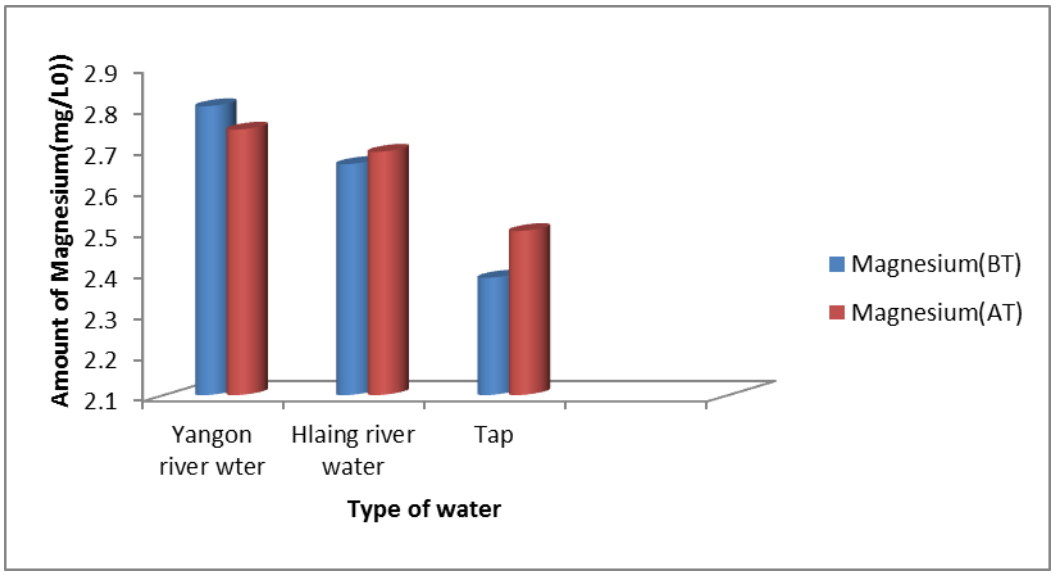

Figure 8- Removal of magnesium from water samples

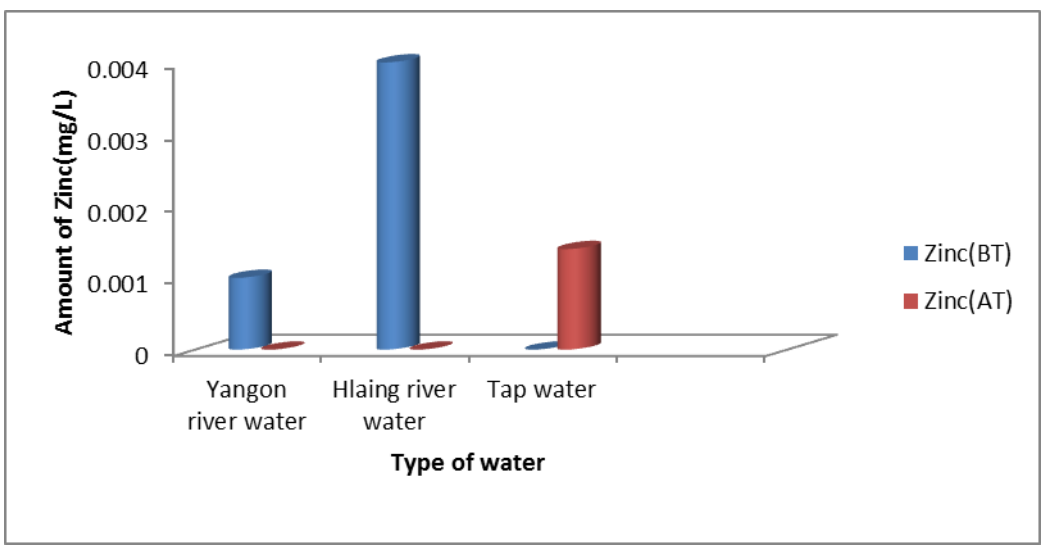

Figure 9- Removal of zinc from water samples

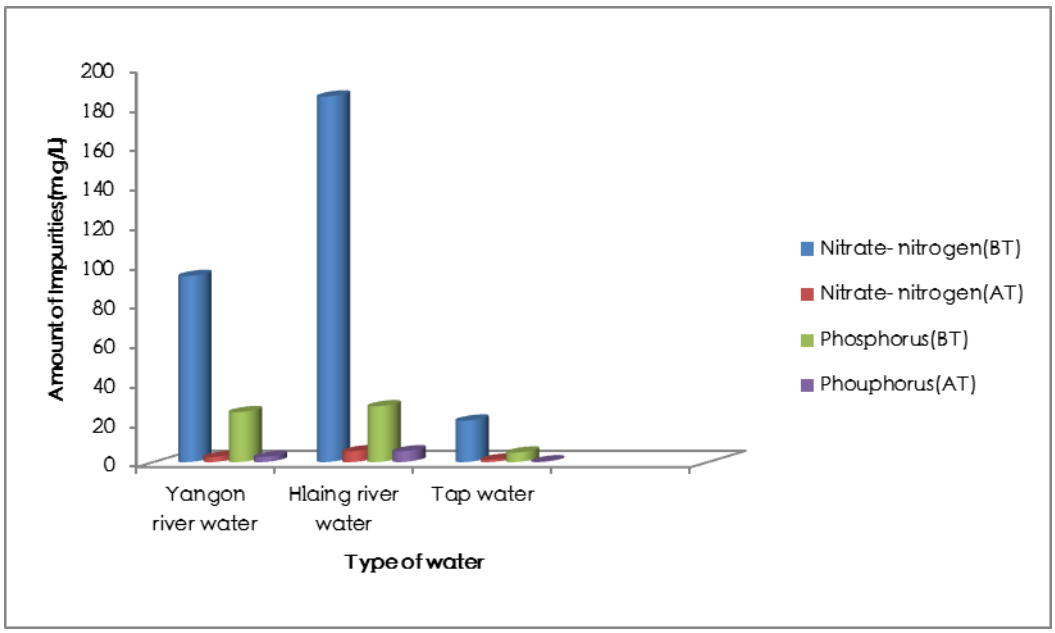

Figure 10 – Removal of nitrate – nitrogen and phosphorous from water samples

Figures 4 to 10 shows the removal of metal (arsenic, calcium, iron, manganese, magnesium, zinc) , nitrate nitrogen and phosphorus from water samples. The filter could remove amount of arsenic less than 0.005 mg/L .The amount of iron could be removed 99.8% from all three water samples. The filter could reduce the amount of calcium and in nearly 20.6% from Yangon river water and Hlaing river water but the amount of calcium was increased 41% after treatment in tap water. It may be due to impurities from adsorbents of filter. The amount of manganese could be removed approximately 90% from all three water samples. It was shown that a little increase in amount of magnesium in Hlaing river water although about 2% of magnesium can be reduced from Yangon river water. The amount of lead present in all three water samples were below the detectable limit of instrument. One major drawback of naturally occurring organic substances as precursors for activated carbon is that the resulting pore size distribution cannot be controlled [8].It could be shown by the above water parameters after treatment with filter.

The present study has revealed that valuable adsorbents could be recovered from coconut shell given the renewable, cheap abundant source of this waste. Result of this study could provide activated carbon consumers with cost effective and environmentally friendly by alternative sources. Activated carbon prepared by sulphuric acid were better adsorbents than those prepared by phosphoric acid and zinc chloride showing the prepared carbons exhibited better characteristics. The variations in characteristics were a function of activation process, activation reagent and activation time.

Ash content indicates the quality of an activated carbon. It is the residue that remains when carbonaceous portion is burned off. The ash consists mainly of minerals such as silica and oxides of aluminum, iron, magnesium, and calcium(oxides). Ash contents of S-1were generally higher than those of others.

The iodine number is the amount of iodine, in milligrams, adsorbed per gram of carbon when the

equilibrium concentration (C_e) of iodine is 0.02M. It has been established that the iodine number in mg/g gives an estimate of the surface area in m2/g[9], and measures the porosity for pores with dimensions between 1.0 – 1.5 nm[10]. The removal of iodine by the activated carbons is related to their porosity characteristics which determine the degree of accessibility of these molecules. A lower iodine number can be ascribed to the presence of pores narrower than 1.0nm, which make up most of the structure of these carbons [11].

It should be noted here that four carbons samples showed (S1 to S4) lower iodine values implying lower surface areas than typical range of 31 to 56 mg/g. AC recommended for water treatment, are to show iodine values ranging from 600 to 1100mg/g[12]. Thus the ACs produced in this study are not advisable for use in water treatment but could show higher efficiency if used for effluent treatment without additional treatment measures.

Bulk density is the mass of carbon that can be contained in a filter of a given solid capacity and the amount of treated liquid that can be retained by the filter cake. The higher the density the better the filterability of activated carbons. However, ACs with bulk density of 0.5g/$cm^3$is adequate for decolourization of sugar[11] According to AWWA, [12], lower limit of bulk density is 0.25g/ $cm^3$for granular activated carbon could be put into practical use[12].The bulk density of all activated carbons were higher than this lower limit, the generally increased from higher soaking time with lower activating time to lower soaking time with higher activating time, activated carbons ranging from S3(0.646) soaking time 7 days with activating time 2 hours to S-7(0.669) soaking time 2 days with activating time 4 hours.

4. CONCLUSION

According to the analysis results, activated carbon obtained from the 10% sulphuric acid solution is the best condition for water treatment and it has a lot of nano pores due to iodine value. The optimum soaking time is 4 days and the optimum activation time is 4 hours. The activated carbon obtained from these conditions can be used to remove some metals from river water and tap water. It can be also used as a colour removal from some liquid chemicals and some beverages.

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