LOW-COST ADSORBENT FOR ARSENIC REMOVAL FROM RURAL WATER SUPPLY SYSTEMS IN NL

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Low-Cost Adsorbent for Arsenic Removal from Rural Water Supply Systems in Newfoundland and Labrador

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Executive Summary

The fly ash from the Corner Brook Pulp and Paper (CBPP) mill was used in this study as the raw material for the preparation of a low-cost adsorbent for arsenic removal from the well water of Bell Island. The CBPP FA was physically activated in two different ways: (a) activation with pure CO_2 (CAC) with the iodine number and methylene value of 704.53 mg/g and 292.32 mg/g, respectively; and (b) activation with the mixture of CO_2 and steam (CSAC) with the iodine number and methylene value of 1119.98 mg/g and 358.95 mg/g, respectively, at the optimized temperature of 850 ºC and the time of 2 hours for both activations. The BET surface area of CAC and CSAC at the optimized conditions was 847.26 m^2/g and 1146.25 m^2/g, respectively. The optimized CSAC was used for impregnation with iron (III) chloride ($FeCl_3$) with different concentrations (0.01 M to 1 M). The study shows that the adsorbent impregnated with 0.1 M $FeCl_3$ is the most efficient adsorbent for arsenic removal. According to the scanning electron microscopy images and BET surface area analysis, it was revealed that impregnation with 0.1 M $FeCl_3$ would not significantly decrease the surface area and pore blockage is also negligible.

Isotherm analysis shows that the Langmuir model will better describe the equilibrium behavior of the arsenic adsorption from both local well water and synthesized water compared to the other models and the maximum arsenic adsorption capacity was 35.6 µg/g of carbon for local well water and 1428.6 µg/g of carbon for synthesized water. Furthermore, the kinetic data of arsenic adsorption from synthesized and local well water was better fitted with the pseudo-second order kinetic model.

Above all, the iron impregnated CBPP AC generated in this research has shown impressive adsorption capacity for arsenic in well water.

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1. Introduction

1.1. Health effects and long-term exposure

Through a long period of exposure to high concentration, arsenic gets accumulated in the human body and cause adverse health effects such as skin lesion, pigmentation changes in skin, and lung, bladder, and skin cancer. Arsenic commonly appears in both organic and inorganic forms in the natural waters. Organic arsenic is less of a concern because it is transformed into nontoxic forms through methylation (Yao et al., 2014).

In water systems, the arsenic usually occurs in arsenate, As (V) and arsenite, As(III) forms (Lorenzen et al., 1995, Rageh et al., 2007). Comparing to arsenic (V), arsenic (III) is more soluble, mobile, and toxic. Long term exposure to inorganic arsenic can significantly increase the risk of different cancers such as skin, lung, liver, bladder, kidney cancer (Rohail, 2012, Yao et al., 2014). Since the drinking water is considered as the major source of exposure to inorganic arsenic, finding a simple, economic, and efficient solution for arsenic removal is critical.

1.2. Source of arsenic contamination in our province

Thirty percent of residents in the rural areas in Newfoundland and Labrador use groundwater and 75% of these wells are private wells (Sarkar et al., 2012, Department of Municipal Affairs and Environment, 2010). The study shows that most of these wells have arsenic level above maximum acceptable concentration (MAC) of 10 µg/L and the background concentration of arsenic in these wells can reach as high as 60 µg/L. The Town of Wabana on Bell Island, shown in Figure -12, have been found previously to contain high level of arsenic in the wells which is 62.0 µg/L (Rohail, 2012).

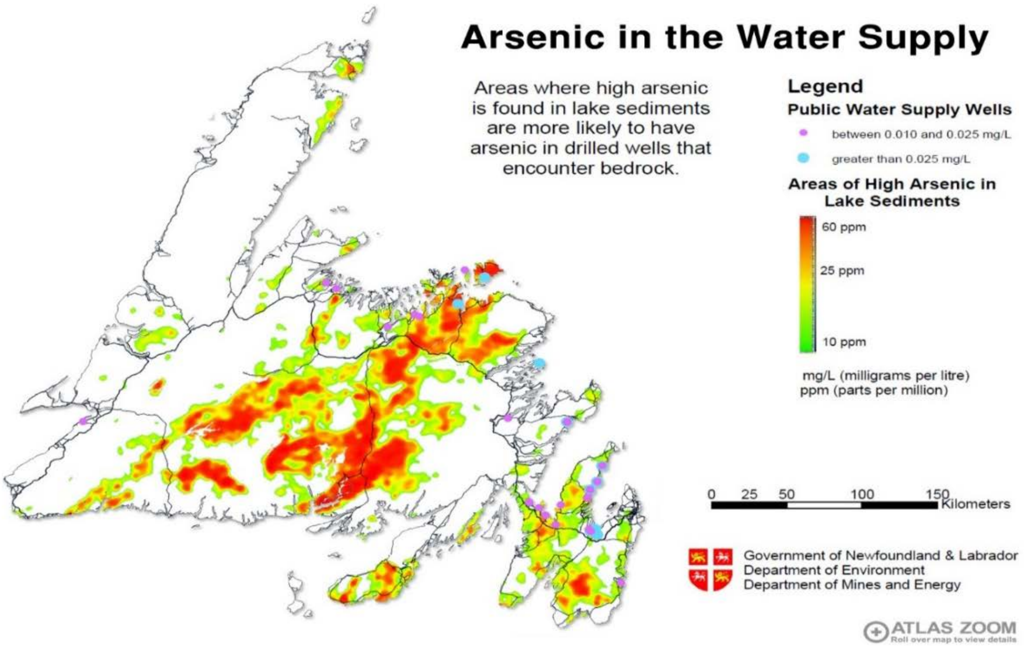

Figure 1-1: Areas of potential arsenic concentration in well water (Department of Municipal Affairs and Environment, 2016)

As a main drinking water source, groundwater, especially well water contaminated by arsenic, has been documented in many communities in Newfoundland and Labrador. Certain systems such as arsenic adsorption package unit have been applied for the water source purification. However, the cost of installation and maintenance is rather high, thus the well owners find it difficult to afford. Besides, some of the techniques are relatively complex and not easy to operate, which can add difficulties to the well owners.

1.3. Current arsenic treatment technologies

The technologies used to treat arsenic from water supply systems are ion exchange, chemical precipitation, electrochemical, reverse osmosis, membrane filtration, floatation and adsorption (Yao et al., 2014, Ray & Shipley, 2015). Among these, adsorption is a simple and efficient method to remove low concentration pollutants. For rural and small communities, it is simple to install and operate and does not require skill operator. Due to high surface area, porous structure, high adsorption capacity, the activated carbon (AC) is proved to be one of the most effective and reliable adsorbent. In order to enhance the removal efficiency of arsenic, synthetically amended activated carbon by coating with metallic compounds has recently gained recognition (Ghanizadeh et al., 2010, Yao et al., 2014). For example, the iron oxide modified AC generated by Yürüm et al. has a maximum adsorption capacity of 27.78 mg/g on arsenic species and the calcium impregnated activated charcoal resulted in 88.09% removal efficiency on the arsenic (III) (Yürüm et al., 2014, Kumar et al., 2013). Iron compounds including hematite, goethite, iron oxide and ferric hydroxide are more preferred to combine with activated carbon due to their high affinity to arsenic adsorption (Zhang & Itoh, 2006, Ghanizadeh et al., 2010).

Some of the commercial activated carbon is usually manufactured using raw materials such as petroleum coke, bituminous and lignite coal, wood products, and coconut shells but due to the high cost of these raw materials, the activated carbon from these materials is not economical and small communities cannot afford (Streat et al., 1995).

1.4. The potential use of carbon extracted from fly ash

The Corner Brook Pulp and Paper (CBPP) mill, located in Western Newfoundland, generates approximate 10,000 metric tons of boiler ash and bottom ash on a yearly basis. This ash is mainly wood ash and is currently dumped into the landfills. The ash from CBPP has very high carbon content (around 90%). It has high pH (above 12) with 80% of CBPP ash has the particle size between 15 and 352 microns (µm) with mean value as 70 µm. According to Chen et al. (2007) and Jahan et al. (2008), a higher micro pore volume could increase not only the iron loading on the AC surface but also the adsorption of arsenic species. From the preliminary study, it is believed that the AC generated from CBPP fly ash has a good potential as an adsorbent by improving its surface using metal oxides (Chen et al., 2007, Jahan et al., 2008).

1.5. Objective and scope of work

The main objective of this study is to develop a metal impregnated activated carbon filter technology by extracting carbon from CBPP ash as this type of carbon is cheaply available and such filters will be easy to install and operate. Commercialized activated carbon products are usually costly due to the high cost of raw materials. Converting the CBPP fly ash into valuable activated carbon product will not only provide affordable adsorbents to rural communities but it will also save money in the CBPP waste management.

2. Preparation and Characterization of the Low-cost Adsorbent

2.1. Methodology

2.1.1 Preparation of CBPP fly ash

CBPP carbon-enriched ash obtained from the Corner Brook Pulp and Paper (CBPP) mill. The CBPP fly ash was first grinded to become powder. The grinded sample before cleaning is named raw CBPP fly ash in this study. Then, it was washed with hot water to remove volatile organic compounds (VOCs) and other impurities, and then it was acid-washed with HNO_3 5% wt/wt to remove metals existed in this fly ash. For the acid wash step, CBPP fly ash and acid was mixed with the ratio 1 g of CBPP fly ash to 10ml of acid and then the mixture was placed on the hot plate at 80 ºC to be heated and rotated for 4 hours.

After that, the mixture was grabbed from the hot plate and placed at room temperature to be cooled down and filtered through 0.45 µm filter paper. In the last step of washing the fly ash, since CBPP was washed with acid, its pH was around 1, so it was needed to be washed with distilled water several times until the pH becomes stable. This step was also done on the hot plate at 80 ºC and mixing the sample with the magnet. After the washing process, the sample was placed in the oven to be dried at 110 ºC overnight. This dried sample, which cleaned with both water and acid, is named cleaned CBPP fly ash in this study.

2.1.2 Characterization of CBPP fly ash

The CBPP fly ash was characterized through the analyzes of particle size distribution, pH value, moisture content, ash content, carbon content, metal content, iodine number and methylene blue value. The analytical method for each characteristic is shown in Table 2-1.

Table 2-1 Characteristics of CBPP fly ash and their corresponding detection methods

Characteristics	**Method of analysis**
Particle size distribution	Horiba Particle Laser Scattered Particles Size Analyzer (Model LA-950)
pH value	ASTM method D3838-05 (2017)
Moisture content	D2867-09 (2014)
Ash content	ASTM method D2866-11 (2011)
Carbon content	Perkin – Elmer 2400 Series II CHN analyzer
Metal content	Perkin – Elmer ELAN DRC II Mass Spectrometer
Iodine number	ASTM method D4607-1 (2014)
Methylene blue value	GB/T 7702.6 (2008)

2.1.3 Activation of CBPP fly ash

Carbonization and activation are the major stages in the activation of raw materials. In the carbonization stage, 10 g of raw materials will be placed in a programmable vertical tube furnace from Carbolite Gero Manufacturer, which provides the versatility and control accuracy to meet the critical temperatures required for the system. The furnace programmed under nitrogen flow (500 cc/min) at 15 ºC/min heating rates until the final temperature was reached. After that, the furnace was kept at the final temperature and under the nitrogen flow of 500 cc/min to complete the carbonization stage.

The activation stage carried out immediately after carbonization, using the CO_2 flow of 500 cc/min and the temperature was kept at final temperature. This activation is called pure CO_2 activation of fly ash. In this study, in order to find out the optimum condition for activation of CBPP fly ash, the effect of both final temperature, and hours of using CO_2 flow were examined. The final temperature of the furnace for activation of CBPP fly ash was changed from 650 ºC to 900 ºC and the CO_2 flow was in the range of 1 to 3 hours.

Moreover, another type of activation was also carried out in this study and it was the activation with the mixture of CO_2 and steam activation. In this type of activation, also, temperature and hours of using gas flow was changed to obtain the optimum condition of activation. Finally, the activated fly ash was cooled down to room temperature and became ready for iron impregnation.

2.1.4 Iron impregnation of activated CBPP fly ash

After activating the CBPP fly ash and cooling it in a desiccator, iron was coated on the activated CBPP fly ash through the impregnation method by using iron (III) chloride ($FeCl_3$) solution, prepared from ferric chloride anhydrous. The procedure of this impregnation is as follows:

1) The activated CBPP fly ash was added to a series of flasks containing $FeCl_3$ solution with different concentrations of $FeCl_3$, from 0 to 1 M, with the ratio of 1 g of activated CBPP fly ash to 20 ml of $FeCl_3$ solution.
2) The samples were mixed with the shaker for 1 hour and the speed of 50 rpm at room temperature.
3) In order to give enough time for iron ions to spread out into the pores of activated CBPP fly ash, mixtures were kept in room temperature for 24 hours.
4) The excess iron solution of each flask was taken out.
5) To impregnate activated CBPP fly ash with iron, the mixture was put in the oven at 110 ºC for 24 hours to start hydrolysis and drying.
6) After drying, the mixture of each sample was washed several times to remove the excess iron of each sample and finally dried 24 hours in the oven at 110 ºC.

2.1.5 Characterization of activated and impregnated CBPP fly ash

i. Scanning electron microscopy (SEM)

The inner faces and surface microstructure of carbon samples before and after activation and after impregnation was observed by using the scanning electron microscopy (SEM) instrument from TERRA facilities.

ii. Iron content

The amount of iron existed in each batch of activated CBPP fly ash could be determined through incineration method (Xu & Teja, 2006), incineration plus acid digestion (Chen *et al.*, 2007), and acid extraction (Gu *et al.*, 2005) which was used in this study. According to this method adsorbents and hydrochloric acid (1:1) were mixed with the ratio of 0.1 gram of adsorbent to 20 ml of acid. Then the mixture was shaken overnight (18 hr) at room temperature with the speed of 120 rpm. After that, mixture was kept at the oven with the temperature of 70 ºC for 4 hours. Finally, the adsorbent was separated from the solution by using the centrifuge with the speed of 6000 rpm for 1 hour and the solution was sent for ICP-OES analysis.

iii. Specific surface area

The specific surface area and porosity analysis of cleaned, activated, and impregnated activated CBPP fly ash were measured at the Centre for Catalysis Research and Innovation (CCRI) in the University of Ottawa. The surface area and pore volume of the samples were determined by N_2 sorption-desorption isotherms at 77 K by applying a 3Flex Surface Characterization Analyzer (Micromeritics Instrument Corporation), following the Brunauer, Emmett, and Teller (BET) method. Fly ash (FA) samples generated by CBPP were collected and delivered to the Environment Laboratory located in the Department of Engineering and Applied Science at Memorial University of Newfoundland.

2.2. Results and discussion

2.2.1 Characterization of raw and clean CBPP fly ash

i. Particle size distribution

According to Figure 2-1, which is the particle size distribution of CBPP fly ash after grinding, the size of the CBPP fly ash particles are from 7.8 to 710 µm. Most of them have the size of between 37 to 300 µm, and about 25% is below 30 µm (Zhang *et al.*, 2017).

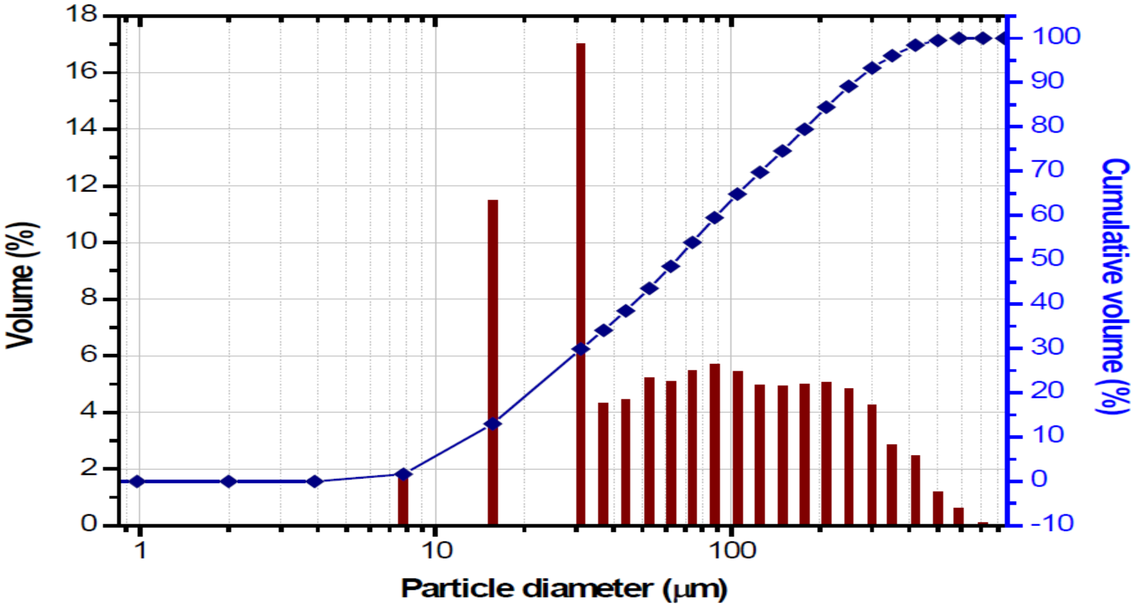

Figure 2-1: particle size distribution of CBPP fly ash after grinding (Zhang et al., 2017)

ii. Ash content, moisture content, carbon content, pH, IN, and MBV

The pH, ash content, moisture content, carbon content, iodine number (IN), and methylene blue value (MBV) of the raw and cleaned CBPP fly ash is reported in Table 2-2. According to these results, the pH of the raw CBPP fly ash is in the alkaline range while using the nitric acid for removing the impurities, has reduced the pH of cleaned CBPP fly ash to the acidic range. Furthermore, the carbon content was increased after cleaning as the result of decreasing the moisture content and ash content. Moreover, after cleaning, methylene blue value was increased and iodine number decreased (Zhang et al., 2017).

Table 2-2: pH, Moisture content, ash content, carbon content, iodine number, and methylene blue value of raw and cleaned CBPP fly ash (Zhang et al., 2017)

Parameters	Raw CBPP fly ash	Cleaned CBPP fly ash
pH	11.44	2.95
Moisture content (%)	1.67	0.35
Ash content (%)	14.04	4.05
Carbon content (%)	78.68	82.79
Iodine number (mg/g)	444.56	57.42
Methylene blue value (mg/g)	57.42	61.89

iii. Metal Content of CBPP fly ash

Different metals existed in the raw and cleaned CBPP fly ash are reported in Table 2-3. According to these results, while high amount of calcium, aluminum, iron, and magnesium are presented in raw CBPP fly ash, the removal rate of these elements after acid washing is 44.2%, 70.3%, 77.7%, and 63.8% , respectively, which means that this acid washing is appropriate and necessary for preparing the fly ash for activation process (Zhang et al., 2017).

Table 2-3: Metal content in raw and cleaned CBPP fly ash (Zhang et al., 2017)

Metal Element	Raw fly ash (ppm)	Cleaned fly ash (ppm)	Removal rate (%)
Magnesium (Mg)	511.65	185.33	63.8
Aluminum (Al)	947.025	281.31	70.3
Iron (Fe)	784.202	175.19	77.7
Zinc (Zn)	11.724	9.07	22.6
Copper (Cu)	7.280	1.75	76
Lead (Pb)	2.252	0	100
Arsenic (As)	< detection limit		
Vanadium (V)	15.460	2.57	83.4
Nickel (Ni)	15.962	3.74	76.6
Calcium (Ca)	2656.356	1481.50	44.2

2.2.2 Activated CBBP fly ash

Before starting any treatment for arsenic removal, preparation of an efficient adsorbent was sufficient and in order to achieve this goal, different parameters were optimized. There are some simple methods for analyzing different types of activated carbons to find out the adsorption capacity, such as methylene blue test and iodine adsorption test, that are usually used for assessing the performance of the adsorbent and we applied them in this study as a rough estimate of adsorption capacity of activated CBPP fly ash also iron impregnated ones. However, to find out the best condition for activation of CBPP fly ash, another parameter, that is the percentage of fly ash burn off during the activation, should also be considered. Thus, the effect of temperature and activation time was examined by using the results of methylene blue value, iodine number, and burn off rate.

i. Effect of activation temperature

In this study CBPP fly ash was activated with pure CO_2 (CAC) at different temperatures: 650 ºC, 700 ºC, 750 ºC, 800 ºC, 850 ºC and 900 ºC and activation time was kept same for activation in different temperatures. The percentage of fly ash burn off, methylene blue value (MBV), and iodine number (IN) of each sample after activation is presented in Table 2-4. According to these data and Figure 2-2, that is the trend of changing iodine number (IN) and methylene blue value (MBV) by temperature, it was found that by increasing the temperature, methylene blue value and iodine number, that are the indicators of mesoporosity and microporosity, respectively, increased.

Thus, the optimum temperature was found as 850 ºC while 900 ºC shows the better result for iodine number and the reason for this recommendation is that by considering the ignition loss at 900 ºC and the energy consumption for activation in this temperature, it seems that activation CBPP at this temperature is not economical while the iodine number and methylene blue value is relatively high at 850 ºC.

Table 2-4: Percentage of fly ash burn off, MBV, and IN of CAC at different temperatures (constant time)(Zhang et al., 2017)

Sample name	Temperature (ºC)	Time (hr)	MBV (mg/g)	Iodine number (mg/g)	Burn-off (%)
CAC	650	1	71.57	529.66	7.79
CAC	700	1	73.84	552.92	14.29
CAC	750	1	76.7	469.26	13.7
CAC	800	1	107.19	502	22.22
CAC	850	1	147.523	515.16	36.84
CAC	900	1	169.33	760.91	53.57

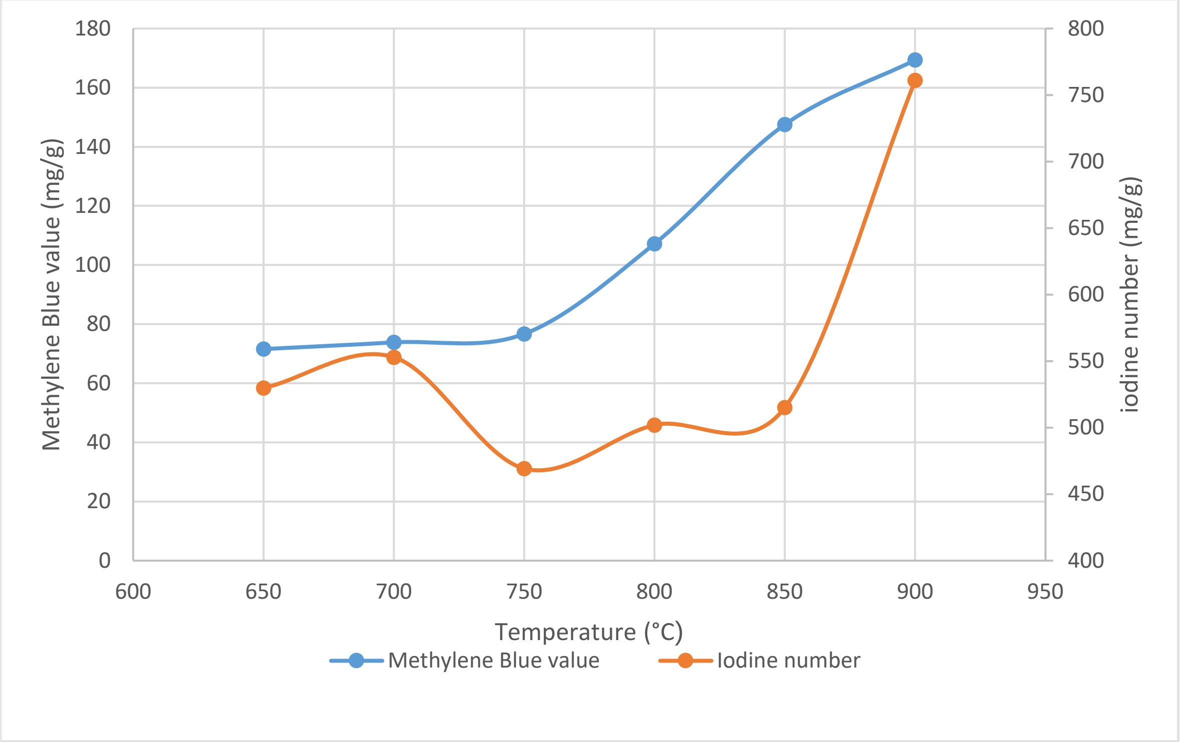

Figure 2-2: MBV and IN of CSA at different temperatures (constant time)(Zhang et al., 2017)

According to these values in Table 2-4, it was decided to run the activation system by applying steam to the system in the last three temperatures that showed higher values of methylene blue and iodine number. Therefore, methylene blue value (MBV), the percentage of fly ash burn-off, and iodine number (IN) of the mixture of CO_2 and steam activated samples (CSAC) are shown in Table 2-5 and Figure 2-3. It is cleared that applying steam to the activation is more efficient in activating the CBPP fly ash compared with pure CO_2 activation. The reason could be the formation of stronger oxygen groups at carbon surface during the CO_2 activation (González *et al.*, 2006).

The highest results related to the 900 ºC but it should be mentioned that it is not cost-effective to activate the CBPP fly ash with the high amount of ignition loss and energy consumption for this activation. Thus, according to these results and figures and also considering the economic aspects, for the mixture of CO_2 and steam activation, also, 850 ºC was accepted as the best temperature for the steam activation due to its high iodine number and methylene blue.

ii. Effect of activation time

After optimizing the activation temperature, CBPP fly ash was activated in different time periods at the optimized temperature. Hence, CBPP fly ash was activated with the mixture of CO_2 and steam (CSAC), and pure CO_2 (CAC) for 1, 2, and 3 hours separately and the results of methylene blue value (MBV), iodine number (IN) and ignition loss for CAC and CSAV are presented in Table 2-6 and Table 2-7, respectively. Figure 2-4 and Figure 2-5 are showing the trend of methylene blue value (MBV) and iodine number (IN) changes with time after activation with pure CO_2 and the mixture of CO_2 and steam, respectively. Thus, CSAC at 850 ºC for 2 hours was selected as the efficient adsorbent with high adsorption capacity due to its high iodine number (IN) and methylene blue value (MBV) for impregnation.

Table 2-5: Percentage of fly ash burn off, MBV, and IN of the CSAC at different temperatures (constant time)

Sample name	Temperature (ºC)	Time (hr)	MBV (mg/g)	Iodine number (mg/g)	Burn-off (%)
CSAC	800	1	120.36	580.14	34.15
CSAC	850	1	234.29	717.73	47.62
CSAC	900	1	256.32	792.56	71.26

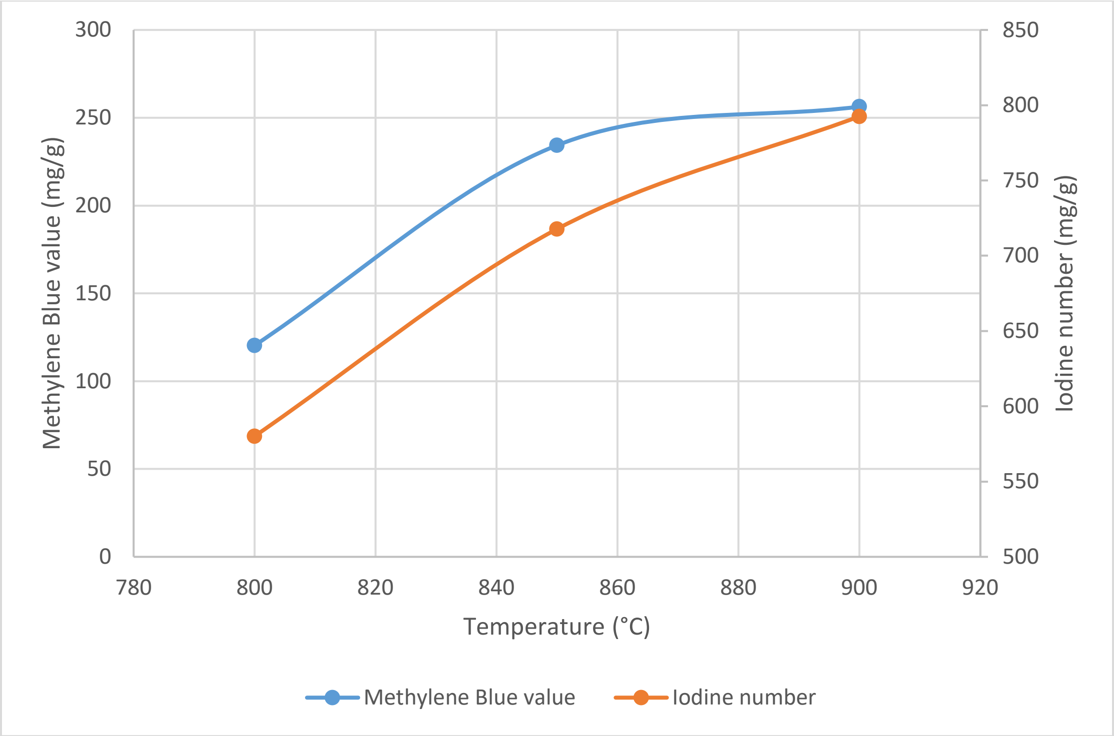

Figure 2-3: MBV and IN of CSAC at different temperatures (constant time)

Table 2-6: Percentage of fly ash burn off, MBV, and IN of CAC at different temperatures (constant time) (Zhang et al., 2017)

Sample name	Temperature (°C)	Time (hr)	MBV (mg/g)	Iodine number (mg/g)	Burn-off (%)
CAC	850	1	147.523	515.16	36.84
CAC	850	2	292.317	704.53	41.12
CAC	850	3	236.094	617.63	65.59

Table 2-7: Percentage of fly ash burn off, MBV, and IN of CSAC at different temperatures (constant time)

Sample name	Temperature (°C)	Time (hr)	MBV (mg/g)	Iodine number (mg/g)	Burn-off (%)
CSAC	850	1	234.29	580.14	32.18
CSAC	850	2	358.95	1119.98	47.62
CSAC	850	3	374.69	1069.92	72.29

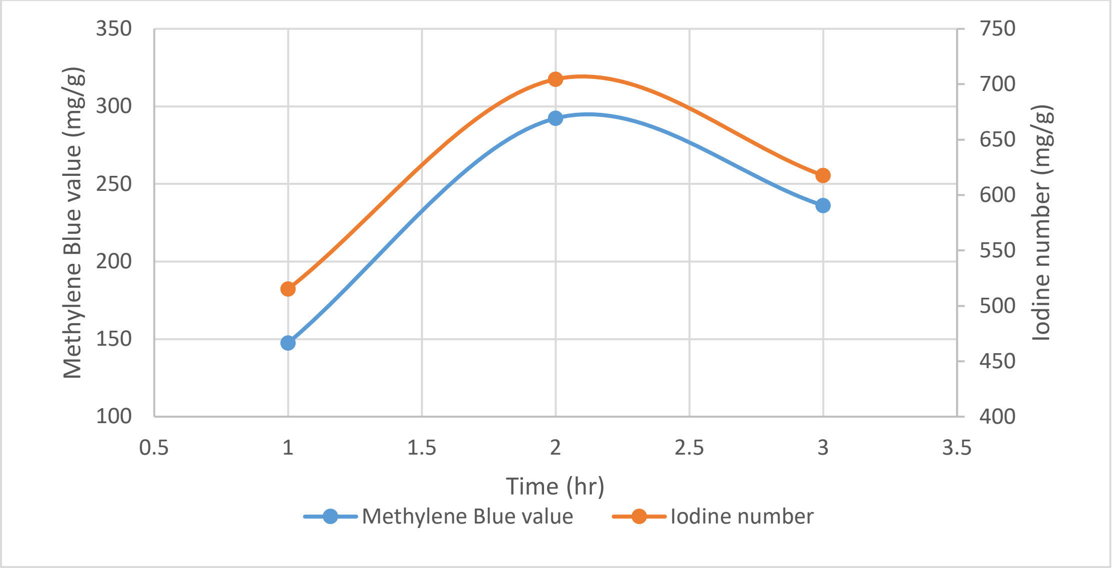

Figure 2-4: MBV and IN changes with time for CAC at 850ºC (Zhang et al., 2017)

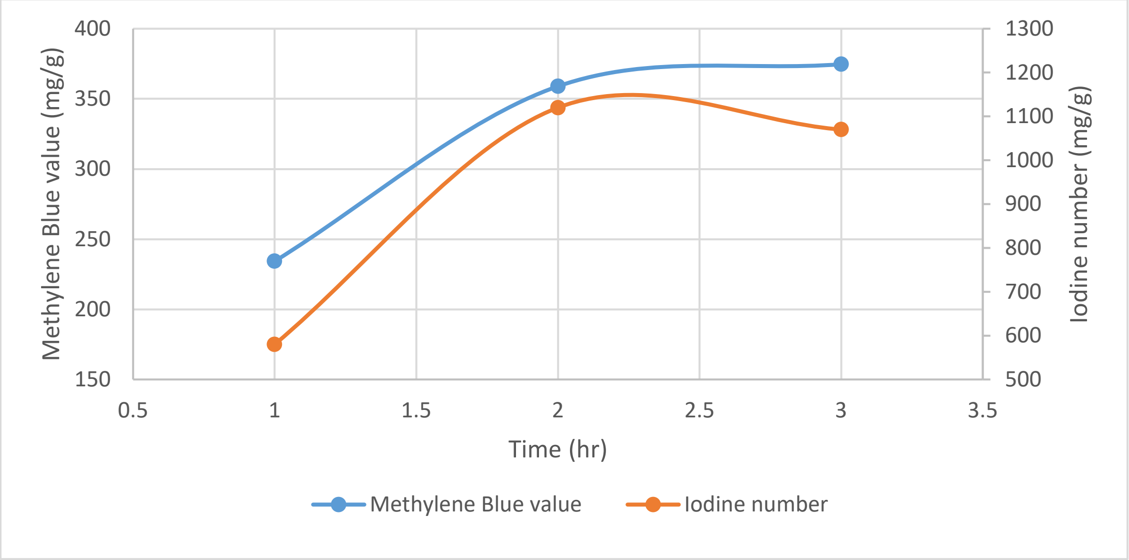

Figure 2-5: MBV and IN changes with time for CSAC at 850ºC

2.2.3 Iron impregnated activated CBPP fly ash

Based on the study conducted by Raychounhury et al. (2015) for removing arsenate and arsenite from drinking water, it was decided to follow their procedure for this study. Hence, activated CBPP fly ash with the mixture of CO_2 and steam (CSAC) was impregnated with iron (III) chloride (anhydrous). Different adsorbents prepared from CSAC samples impregnated with different concentrations of iron chloride from 0.01 M to 1 M. In order to find the best concentration to apply for impregnation of CSAC for arsenic removal from rural water, each sample after impregnation was tested for arsenic removal from synthesized water with the concentration of 1ppm of sodium arsenate ($Na_2HAsO_4.7H_2O$) or arsenic (III) oxide (As_2O_3). The percentage of arsenic (V) removal for samples impregnated with different iron chloride concentration is shown in Table 2-8 and also it is plotted in Figure 2-6.

Moreover, according to the ICP-MS results of the treated water with each of these impregnated adsorbents, it was observed that for first 4 samples (impregnated with iron chloride concentrations between 0.3 M to 1 M), even after washing for several times, a considerable amount of iron leached to the water in high concentrations. Hence, it was concluded that samples impregnated with iron chloride with concentration 0.01 M to 0.2 M would give better results in arsenic removal without iron leaching in water. Therefore, these samples were tested to treat synthesized water contaminated with arsenic (III) oxide (As_2O_3) and the percentage of arsenic (III) removal by using these adsorbents is reported in Table 2-9 and Figure 2-7.

Based on the results listed in tables and figures above, it is clear, that the best concentration of iron (III) chloride for CBPP fly ash impregnation is 0.1 M, as this concentration showed the most efficient ability in arsenic removal from water. Hence, this modified adsorbent was used to treated the local well water of Bell Island with the ratio of 0.1 g of adsorbent to 200 ml of well water and the concentration of different elements are presented in Table 2-10.

Table 2-8: Percentage of arsenic (V) removal for CSAC samples impregnated with different iron chloride concentration

Sample#	Iron chloride concentration for impregnation (M)	% removal of As(V)
1	1	98.34
2	0.5	98.13
3	0.4	98.44
4	0.3	98.08
5	0.20	99.42
6	0.10	99.63
7	0.05	98.63
8	0.02	97.62
9	0.01	94.11
10	0	80.43

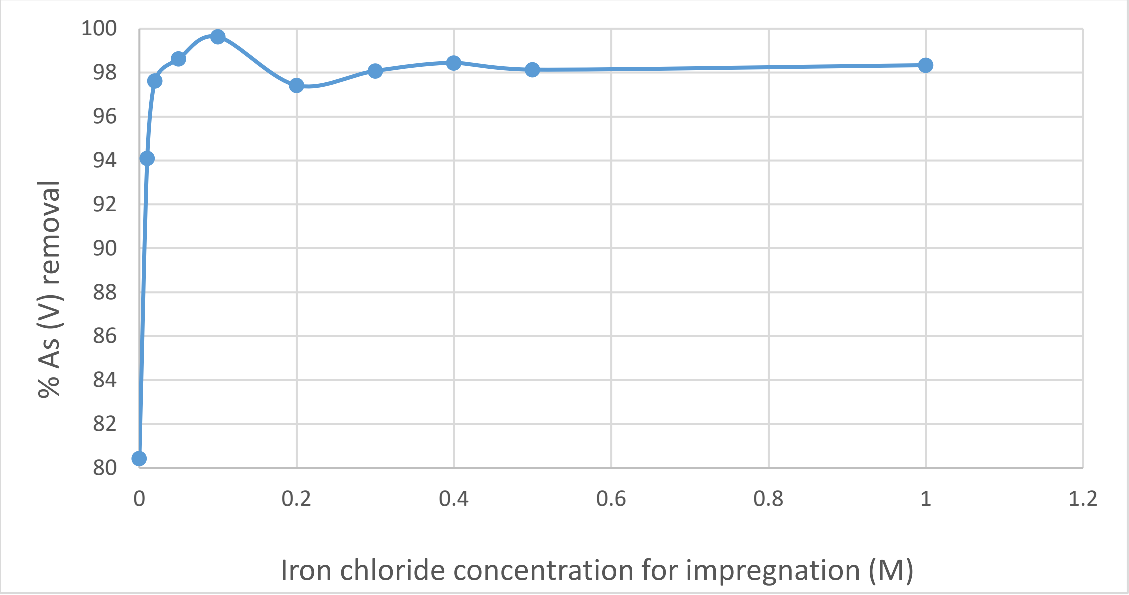

Figure 2-6: Percentage of arsenic (V) removal using CSAC samples impregnated with different iron chloride concentration

Table 2-9: Percentage of arsenic (III) removal using CSAC samples impregnated with different iron chloride concentration

Sample#	Iron chloride concentration for impregnation (M)	% removal of As (III)
1	0.01	81.45
2	0.02	82.84
3	0.05	84.51
4	0.075	85.53
5	0.1	86.64
6	0.2	85.52

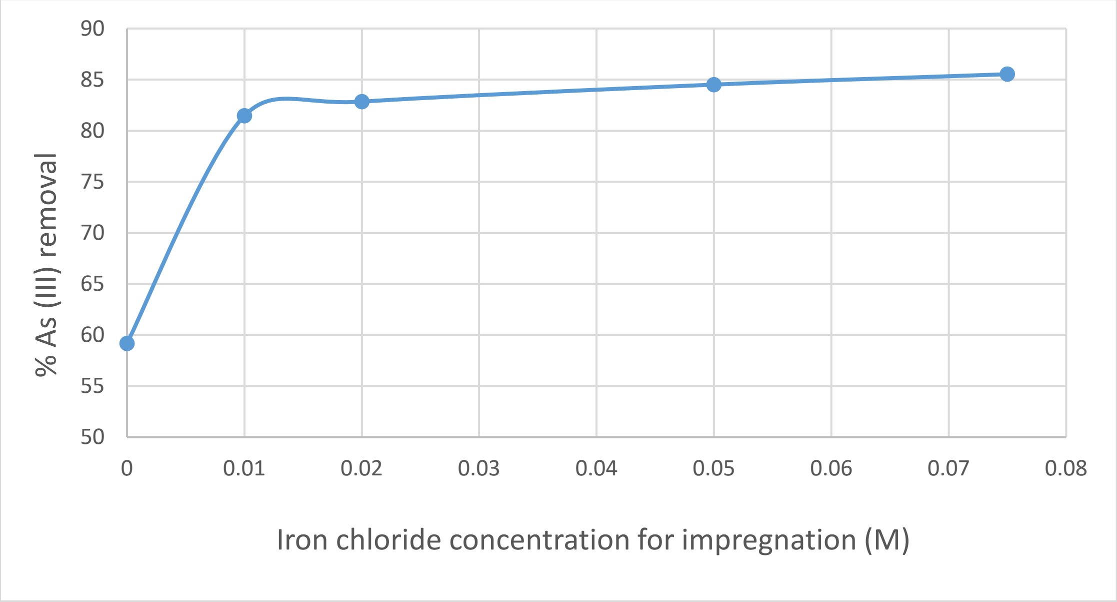

Figure 2-7: Percentage of arsenic (III) removal using CSAC samples impregnated with different iron chloride concentration

Table 2-10: Concentration of different elements in local Well water of Bell Island before and after treatment (with 0.1 M iron impregnated CSAC)

Element	Concentration in well water (ppb)	Concentration after treatment (ppb)
Li	15.3	13.1
Be	0.342	<DL
Pb	0.175	0.03
P	365	<DL
Ti	0.6	0.314
Cr	2.48	<Dl
Mn	256	76.7
Fe	50	<Dl
Cu	4.24	6.13
As	15.7	3.7
Br	50.5	47.7
Zn	<DL	<DL
Sr	340	276
Ni	0.102	<DL
I	1.64	1.17
V	<DL	<DL
Ba	53.1	31.8
Al	11.7	13.3
Mg	5974	5076
Si	6939	6405
Cl	18404	25236
Ca	32126	26236
Rb	1.35	1.24
Cs	0.035	0.032
Be	0.342	<DL
Ce	0.1	<DL

As shown in Table 2-10, applying this adsorbent for the treatment of Bell Island's well water, could not only reduce the arsenic from the water, but also it is capable to reduce the concentration other metals in water except for Cu, Al, and Cl. Concentration of Cl was slightly increased after treatment because the adsorbent was prepared by impregnation with iron (III) chloride and Cl could leach during the treatment. Hence, it is concluded that this method for preparation of the adsorbent is good for arsenic and other metals removal from drinking water and the adsorbent prepared by using 0.1 M of iron chloride solution for impregnation of activated CBPP fly ash was used for other experiments and analyses.

2.2.4 Iron content and SEM images

The SEM images of carbon samples before and after activation, Figure 2-8, reveal that this type of activation with the mixture of CO_2 and steam (CSAC) could produce more pores, especially micropores on CBPP fly ash that is led to achieving an adsorbent with high surface area and it is in good agreement with the values of iodine number and methylene blue.

Furthermore, according to the SEM images of the impregnated samples in Figure 2-8, by increasing the iron content, more pore blockage occurred, and for higher iron concentrations clusters of iron particles were presented on the surface of the sample, that reduce the surface area of the adsorbent. For higher contents of iron, the surface of the adsorbent is covered by the iron particles and then, more iron particles accumulate on the iron particles attached to the adsorbent and these accumulated particles are not attached strong enough and leach to the solution through the adsorption process.

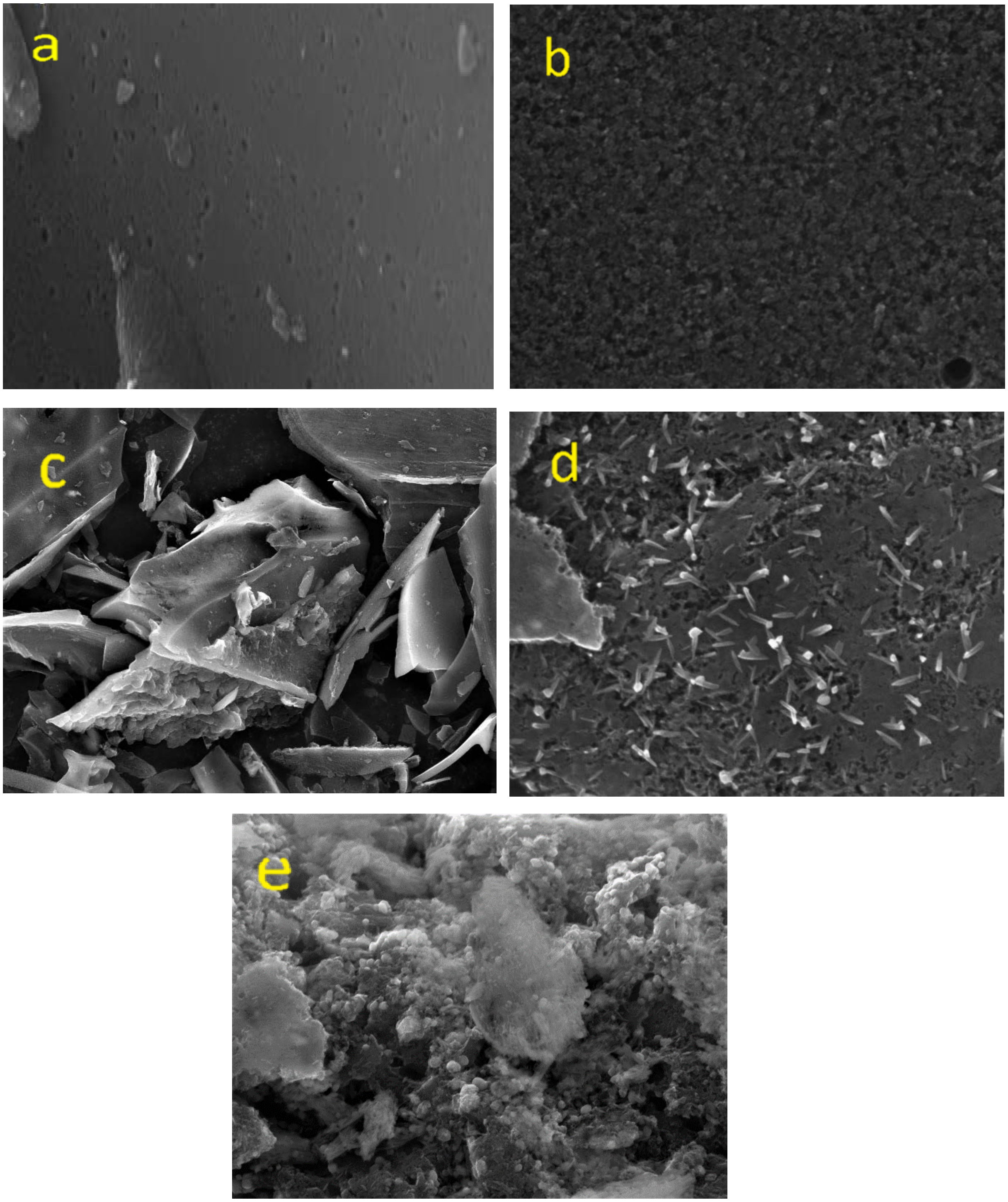

Figure 2-8: SEM image of carbon samples: a) CBPP fly ash before activation, b) CSAC after activation at 850ºC, c) CSAC impregnated with 0.01M $FeCl_3$, d) CSAC impregnated with 0.1M $FeCl_3$, e) CSAC impregnated with 1M $FeCl_3$

After separation of adsorbents from the acid solution, the amount of iron in the solutions was determined with ICP-OES. Concentration of iron in each of these solutions, which are prepared by acid extraction of the iron impregnated samples with concentrations from 0.01 M to 1 M of iron (III) chloride, are shown in Table 2-11.

According to the Table 2-11, and the SEM images of different adsorbents presented above, It is concluded that by increasing the amount of iron, the distribution of iron impregnated on the carbon sample changes from uniform to uneven and nonuniform that causes the pore blockage, reduction of surface area, and iron leaching for higher iron contents during the adsorption process for arsenic removal.

Iron content for the most efficient adsorbent for arsenic removal, that was determined in the previous section and it is the sample impregnated with iron chloride with the concentration of 0.1M, is 1.97%. This modified adsorbent was used for the kinetic and equilibrium experiments.

Table 2-11: Iron content and concentration of impregnated CSAC with different concentrations of $FeCl_3$ from 0.01 M to 1 M

Sample name	**Iron concentration (ppm)**	**Iron content (%)**
Impregnated with 0.01 M $FeCl_3$	4.67	0.19
Impregnated with 0.02 M $FeCl_3$	6.21	0.25
Impregnated with 0.05 M $FeCl_3$	19.26	0.77
Impregnated with 0.1 M $FeCl_3$	49.10	1.97
Impregnated with 0.2 M $FeCl_3$	158.53	6.31
Impregnated with 0.3 M $FeCl_3$	244.94	9.71
Impregnated with 0.4 M $FeCl_3$	336.80	13.50
Impregnated with 0.5 M $FeCl_3$	435.03	17.28
Impregnated with 1 M $FeCl_3$	631.93	25.17

2.2.5 Surface area and pore volume

According to the BET results reported in Table 2-12, on the surface area and porosity of the cleaned, pure CO_2 activated CBBP fly ash (CAC), mixture of CO_2 and steam activated CBPP fly ash (CSAC) , and impregnated CSAC with 0.1 M $FeCl_3$ solution, which was revealed that it is the most efficient adsorbent for arsenic removal in section 2.2.3, it was found that while activation of CBBP fly ash with both pure CO_2 and mixture of CO_2 and steam improve the surface area and micro porosity of the CBPP fly ash, using the mixture of steam and CO_2 in activation increases the surface area more effectively than using the pure CO_2. Moreover, according to the Table 2-12, the impregnation of the steam and CO_2 activated CPP fly ash with 0.1 M $FeCl_3$ would not significantly decrease the surface area, and pore blockage is negligible.

N_2 adsorption- desorption isotherms are plotted for the cleaned, the pure CO_2 activated, the mixture of CO_2 and steam activated, and the impregnated CBPP fly ash in Figure 2-9, Figure 2-10, Figure 2-11, and Figure 2-12, respectively. It is obvious from these plots that the adsorption rate of activated samples, especially sample activated with the mixture of CO_2 and steam, is significantly increased compared to the cleaned carbon and it is in a good agreement with the methylene blue and iodine number results. About the N_2 adsorption- desorption isotherm of the impregnated sample, Figure 2-12, it is again revealed that the impregnation of the steam and CO_2 activated CPP fly ash with 0.1 M $FeCl_3$, did not decrease the adsorption rate significantly compared to the activated CBPP fly ash with the mixture of steam and CO_2 while it is still notably higher than the cleaned and pure CO_2 activated samples.

Table 2-12: Surface area and pore volume of cleaned CBPP fly ash, CAC, CSAC, and impregnated CSAC

CBPP Sample	**Surface area (m^2/g)**	**Micropore area (m^2/g)**	**Pore volume (cm^3/g)**
Cleaned	486.44	402.5	0.18
2 hours pure CO_2 activated @850ºC	847.26	619.49	0.28
2 hours (Steam+CO_2) activated @850ºC	1146.25	648.90	0.29
2 hours (Steam+CO_2) activated @850ºC-impregnated with 0.1M $FeCl_3$	1074.45	572.84	0.26

Note: The results of surface area, micropore area, and pore volume of cleaned and 2 hours pure CO_2 activated CBPP fly ash at 850 ºC was obtained from the report of Zhang et al. (2017).

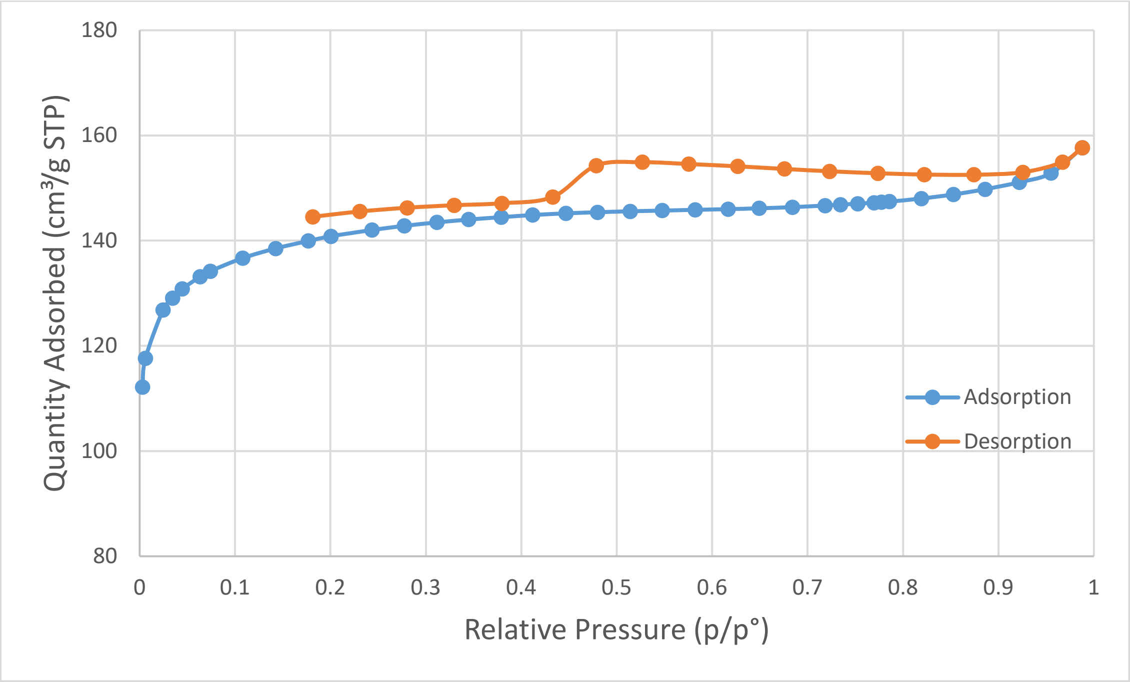

Figure 2-9: N_2 adsorption-desorption isotherm linear plot of raw and cleaned CBPP fly ash (Zhang et al., 2017)

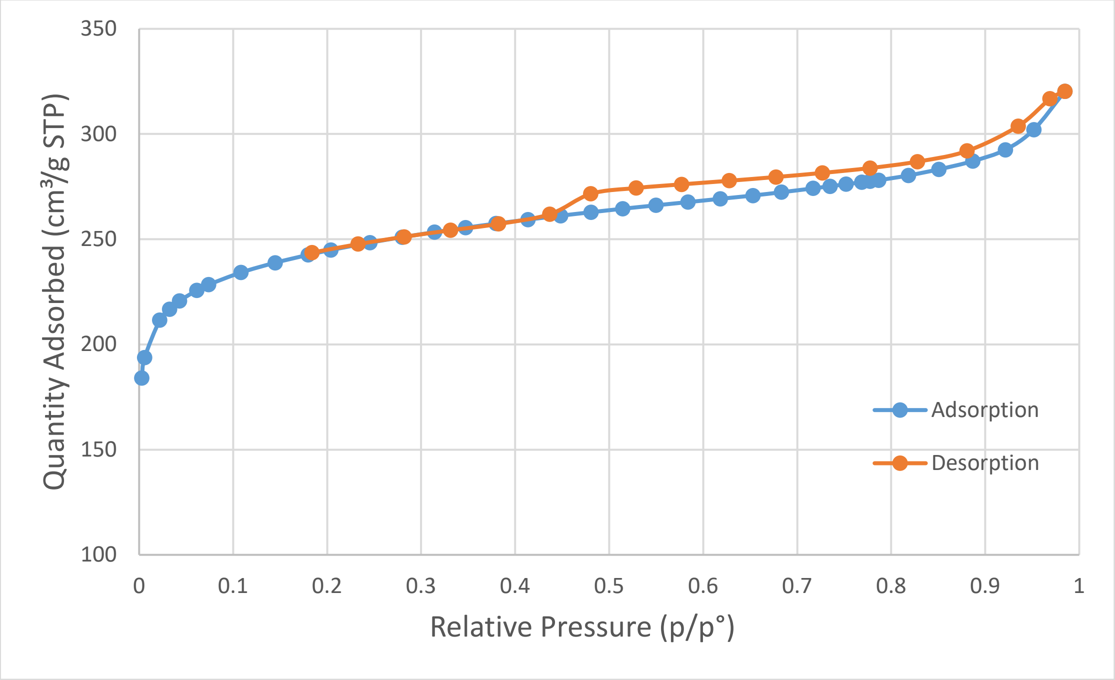

Figure 2-10: N_2 adsorption-desorption isotherm linear plot of CSA (Zhang et al., 2017)

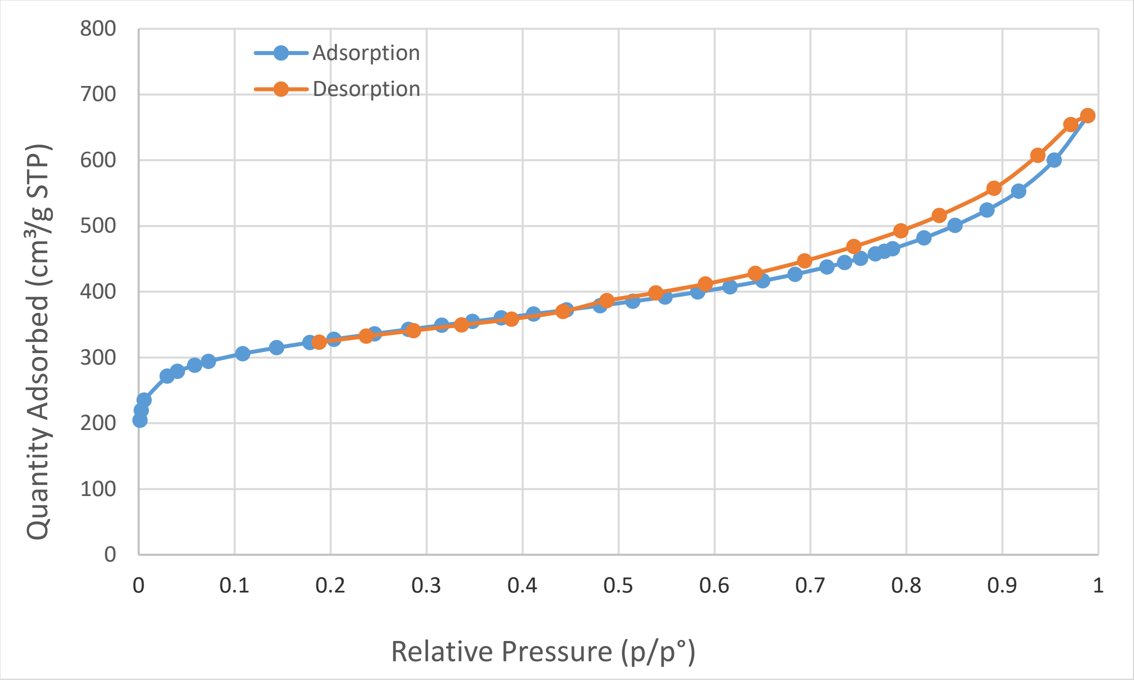

Figure 2-11: N_2 adsorption-desorption isotherm linear plot of CSAC

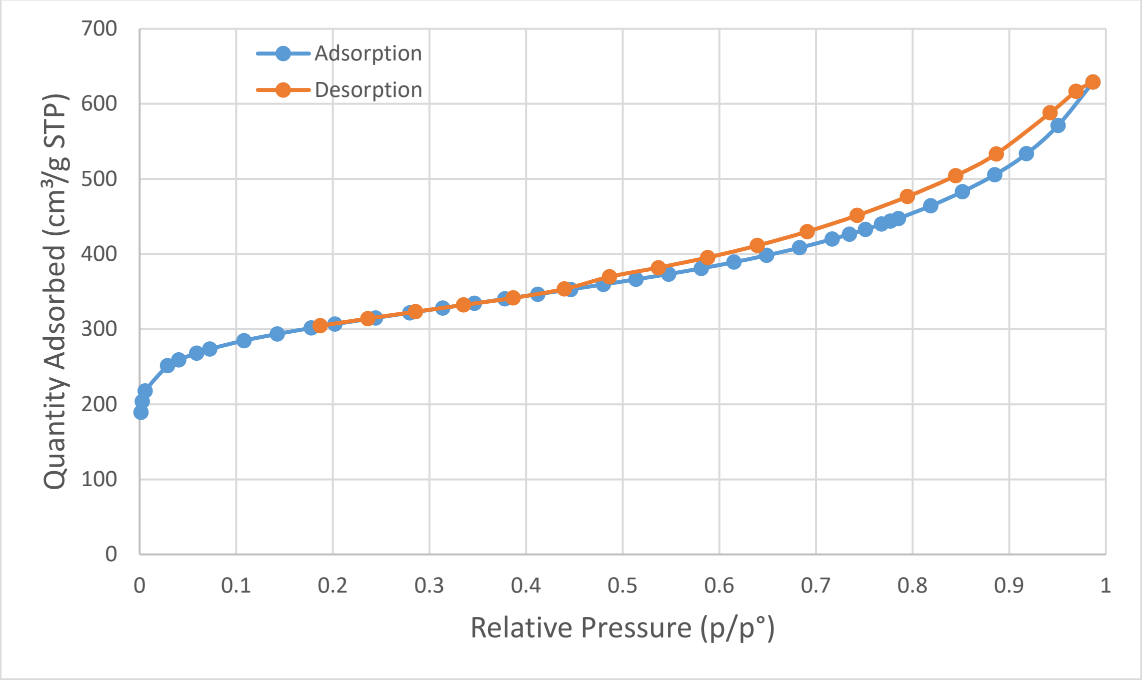

Figure 2-12: N_2 adsorption-desorption isotherm linear plot of CSAC impregnated with 0.1 M $FeCl_3$

3. Sorption Experiments on Arsenic-Contaminated Water

3.1. Methodology

3.1.1 Elemental analysis of Bell Island's well water

The arsenic contaminated well water in Bell Island was collected and in order to find out different elements existed in this well water and their concentrations, it was sent to be analyzed by the Inductively Coupled Plasma Mass Spectrometry (ICP-MS) device.

3.1.2 Arsenic removal experiment

The developed absorbent was then applied to measure the performance of arsenic removal from synthetic water. All chemicals used for the solutions were reagent grades in distilled water with electrical conductivity (EC) less than 3 μmohs/cm. The stock solution of arsenate, As(V), and arsenite, As(III), were prepared from sodium arsenate ($Na_2HAsO_4.7H_2O$) and arsenic (III) oxide (As_2O_3), respectively, with concentration of 1000 mg/L. For this experiment, the stock solutions were diluted to the concentration of 1 mg/l (ppm). The next step was the batch experiments that were continued by using 0.1 g of the developed adsorbents, impregnated with different concentration of iron solution, in a series of glass containers that each contained 200 ml of As(V) or As(III) solutions. By using an end-over-end rotator, containers were mixed for 24 hours at room temperature.

Finally, samples were filtered through the 11cm filter paper and sent for ICP-MS analysis. Activated CBPP fly ash, impregnated with different concentrations of $FeCl_3$ from 0.01 M to 1 M and each of these impregnated adsorbents were used for arsenic removal through this procedure and under the same conditions in order to find out the efficiency of these adsorbents and finally to determine the efficient one. The efficient impregnated activated CBPP fly ash, which is the sample impregnated with 0.1 M $FeCl_3$, was used for sorption kinetic tests and equilibrium sorption experiments.

3.1.3 Sorption kinetic test

The adsorption of arsenic from both local well water of Bell Island and synthesized water, on the prepared iron impregnated activated CBPP fly ash was investigated and the efficient impregnated activated CBPP fly ash was used for sorption kinetic experiments. For these experiments, also, synthesized water with concentration of 1 ppm was prepared by using sodium arsenate ($Na_2HAsO_4.7H_2O$). For kinetic sorption tests, 200 ml of local well water (or synthesized water) was added to a series of glass bottles that contained 0.1 g of the developed and efficient adsorbent. Each of these bottles, then, was mixed by an end-over-end rotator at room temperature for specific time, from 5 minutes to 24 hours to ensure that the equilibrium was reached for the adsorbent and arsenic contaminated water. All samples were then filtered through the 11cm filter paper and sent for ICP-MS analysis.

3.1.4 Equilibrium sorption experiments

The equilibrium sorption experiments were also conducted for both synthesized water with the concentration of 1 ppm prepared by using sodium arsenate ($Na_2HAsO_4.7H_2O$) and local well water of Bell Island. To find out the equilibrium condition and sorption isotherm, a series of glass bottles, with the amount of 0.1 g of the efficient adsorbent from CBPP fly ash inside, was prepared. Then, from 50 ml to 1000 ml of arsenic contaminated local well water (or synthesized arsenic contaminated water) added to these bottles and mixed for 24 hours at room temperature with an end-over-end rotator. All samples were then filtered through the 11cm paper and sent for ICP-MS analysis. The results were then compared with different models to find the best model fitted with these results.

3.2. Results and discussion

3.2.1 Elemental analysis of Bell island's well water

Water samples from the well water of Bell Island analyzed for metal concentration using ICP-MS and the results are reported in Table 3-1. According to these results, the concentration of arsenic in this water is higher than the maximum acceptable concentration of arsenic which is 10 µg/L and the treatment is required for this water prior to being used. Moreover, the existence of other elements, with relatively high concentrations, are representing that the arsenic adsorption capacity of the modified adsorbent in this study would be lower than the other adsorbents reported in other studies applied synthesized water contained arsenic. Moreover, pH of this water was also determined and it was 7.32.

Table 3-1: Concentration of different elements existed in the raw Bell Island’s well water

Element	Concentration (ppb)	Element	Concentration (ppb)
Li	15.3	Rb	1.35
Be	0.342	Sr	340
Pb	0.175	Mo	0.3
P	365	I	1.64
Ti	0.600	Cs	0.032
Al	11.7	Ni	0.102
Cr	2.48	Ba	53.1
Mn	256	Ce	0.1
Fe	50	Mg	5974
Cu	4.24	Si	6939
As	15.7	Cl	18404
Br	50.5	Ca	32126

3.2.2 Equilibrium sorption isotherms

To understand the mechanism of the adsorption process, sorption isotherms are essential. Sorption isotherms give an equilibrium relationship of arsenic concentration between liquid phase and adsorbents which are the solid phase at a constant temperature. The results achieved from the equilibrium sorption experiments of arsenic removal from the local well water and synthesized water with $Na_2HAsO_4.7H_2O$ (1 ppm), using the CSAC sample impregnated with the 0.1 M $FeCl_3$ solution, are presented in Figure 3-1 and Figure 3-2, respectively. According to the previous studies on arsenic removal using the adsorption method, Freundlich, Langmuir, and Temkin adsorption isotherm models were commonly used to introduce the mechanism of arsenic adsorption and these models were also examined in this study (Ananta et al., 2015, Dehghani et al., 2017). Different parameters of these models calculated and are reported in Table 3-2.

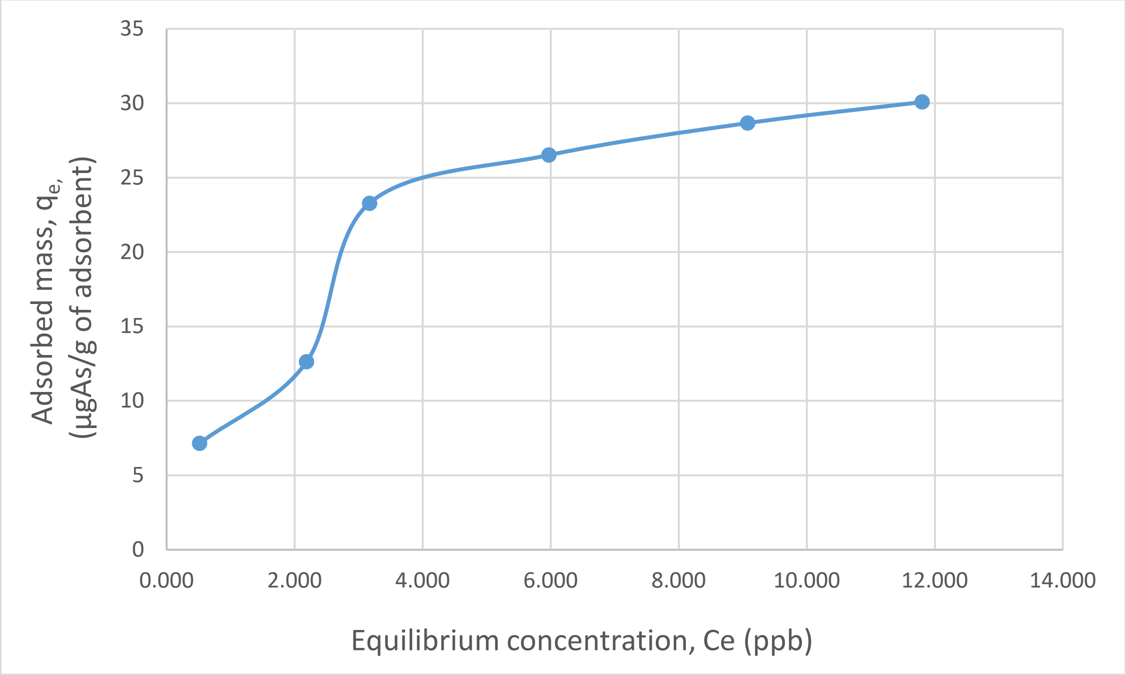

Figure 3-1: Isotherm curve of arsenic removal from local well water

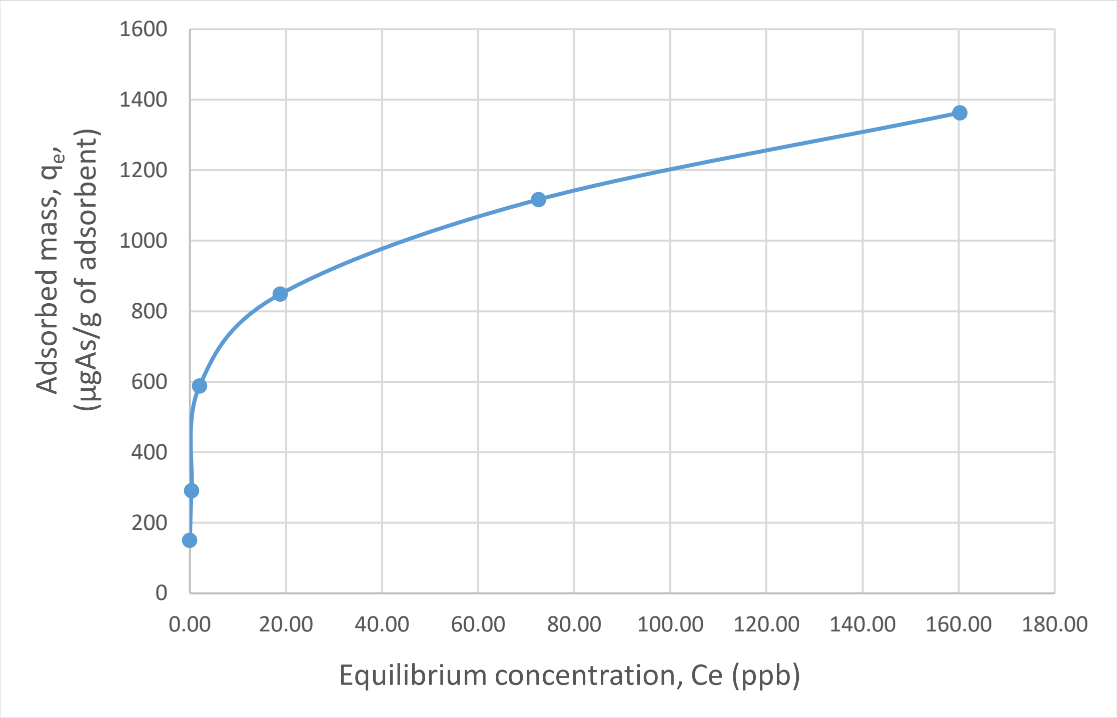

Figure 3-2: Isotherm curve of arsenic removal from synthesized water ($Na_2HAsO_4.7H_2O$, 1 ppm)

Table 3-2: The parameters of Freundlich, Langmuir, and Temkin models for arsenic removal from local well water and synthesized water ($Na_2HAsO_4.7H_2O$, 1 ppm)

Isotherm model	Parameter	Value for local well water	Value for synthesized water
Freundlich	K_f	10.22	413.5
	n	2.07	3.92
	R^2	0.93	0.96
Langmuir	K_L	0.482	0.18
	q_{max}	35.46	1428.6
	R^2	0.997	0.99
Temkin	K_L	0.482	0.18
	q_{max}	35.46	1428.6
	R^2	0.997	0.99

Among the different isotherm models fitted in this study, it is obvious that the Langmuir model is the best model for describing the equilibrium behavior of arsenic adsorption by iron impregnated CSAC ($R^2 \geq 0.99$) for both local well water and synthesized water with $Na_2HAsO_4.7H_2O$ (1 ppm) indicating that the monolayer of arsenic ions covers the adsorbent surface. Hence, according to the results of Langmuir fitting, maximum adsorption capacity of this modified adsorbent in removing the arsenic was 35.46 µg/g from the local well water and 1428.6 µg/g from synthesized water with $Na_2HAsO_4.7H_2O$ (1ppm) and it is in good agreement with other low-cost adsorbents reported in some papers while they usually used only the synthesized water with only arsenic as the constituent (Yadav *et al.*, 2014).

Furthermore, the maximum iron use efficiency or the maximum adsorption capacity with respect to iron is 1.8 mg/g Fe and 72.67 mg/g Fe in removing the arsenic from the local well water and synthesized water with $Na_2HAsO_4.7H_2O$ (1 ppm), respectively. It should be considered that these values are only the maximum adsorption capacity for arsenic and for the local well water, which has a wide range of elements with different concentrations that affect and decrease maximum arsenic adsorption capacity, it would be a competition between elements presented in the well water to reach to the active sites of the adsorbent surface and this is the reason of having lower adsorption capacity compared with adsorption capacity of synthesized water with $Na_2HAsO_4.7H_2O$ (1 ppm) in the present study and values reported in other papers.

In addition, according to the results of other studies for arsenic removal, it was found that increasing the pH from 3 to 7 would not significantly affect the arsenate removal and for arsenite, also, the optimum pH was reported from 6 to 9.5. Hence, the optimum pH, in this study, for adsorption of arsenate and arsenite was set between 6-7, at which arsenate existed mainly as its active species $H_2AsO_4^-$ and $HAsO_4^{2-}$ and arsenite is mostly as un-dissociated species, and also recommended in other studies (Di Natale *et al.*, 2009, Gu *et al.*, 2007, Li *et al.*, 2014, Raychoudhury *et al.*, 2015).

3.2.3 Sorption Kinetics

Sorption kinetics are usually used to examine the adsorption behavior and mechanism and, to find out the steps controlling the reaction rate. Moreover, by using the kinetic models, it is possible to find out the equilibrium time of the reaction (Ho & McKay, 1998). The sorption kinetic results of arsenic removal, using the CSAC impregnated with 0.1M $FeCl_3$, from synthesized water with $Na_2HAsO_4.7H_2O$ (1 ppm) and the local well water were determined in a time zone of 5 minutes to 24 hours and shown in Figure 3-3 and Figure 3-4, respectively. According to Figure 3-3, after 300 minutes, arsenic adsorption on the iron impregnated CSAC reached to the equilibrium, since after 300 minutes the adsorbed mass of arsenic per gram of adsorbent remained constant. For synthesized water, also, it seems that after 20 hours the adsorption of arsenic on the modified adsorbent was reached to the equilibrium.

Pseudo first and pseudo second order kinetic models are usually used for kinetic investigations on activated carbons. Besides, for the porous adsorbents, the diffusional effects are also important, so the mass transfer kinetic models, such as intraparticle diffusion model (the Weber and Morris model) are also applied (Tsibranska & Hristova, 2011). To find out the best model to describe the kinetic of arsenic removal from synthesized water with $Na_2HAsO_4.7H_2O$ (1 ppm) and the local well water, pseudo-first order, pseudo-second order, and intra particle diffusion kinetic models, which used commonly for sorption processes was investigated in this study (Ananta et al., 2015, Chammui *et al.*, 2014). Different parameters of these models calculated and are reported in Table 3-3. Based on the results achieved from Table 3-3, it is obvious that, among the kinetic models investigated in this study, pseudo second order kinetic model has the higher correlation coefficient (R^2) and these kinetic data follow the second order kinetic model better than the other models.

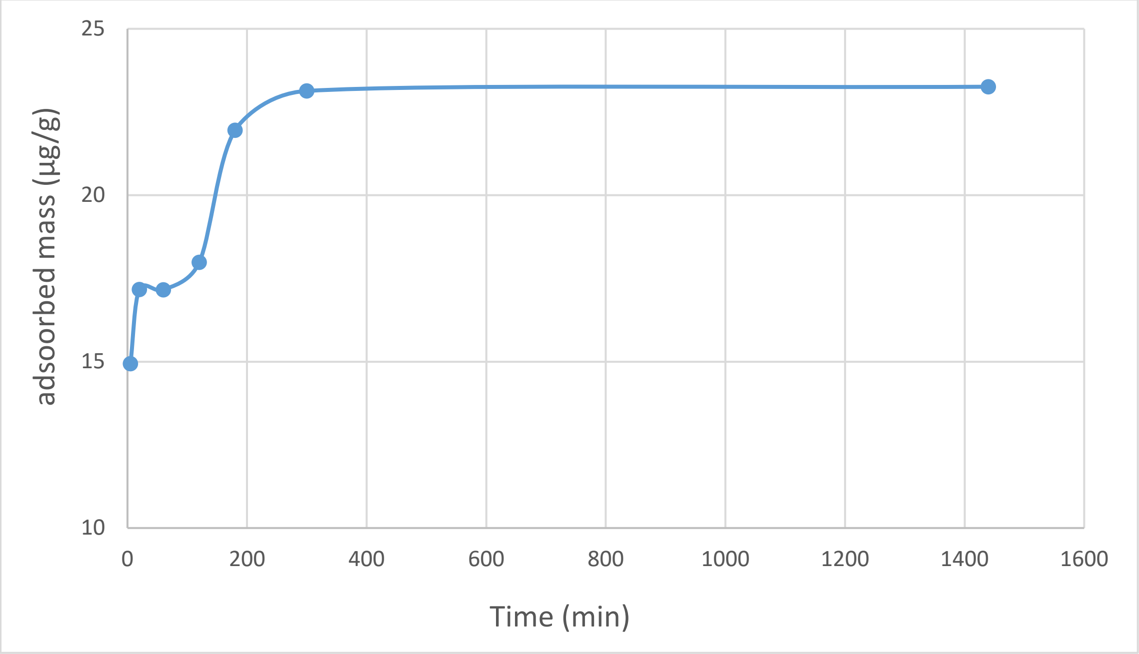

Figure 3-3: Sorption kinetic of local well water

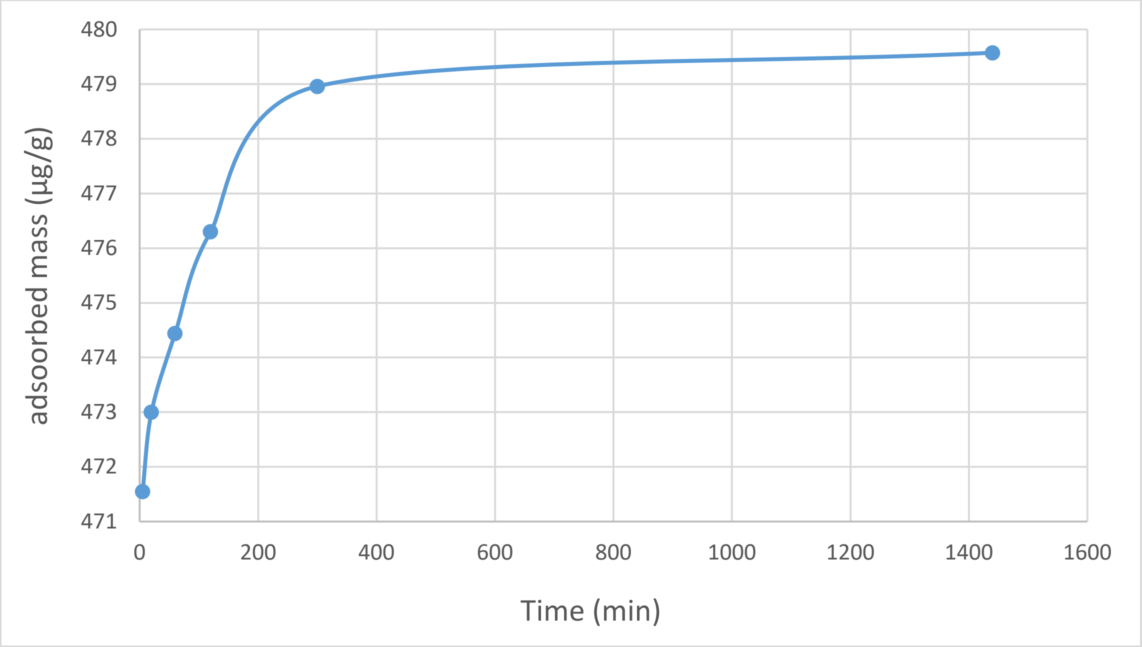

Figure 3-4: Sorption kinetic of synthesized arsenic contaminated water ($Na_2HAsO_4.7H_2O$, 1 ppm)

Table 3-3: Parameters of pseudo-second order, pseudo-first order, and intra particle diffusion kinetic models for local well water and synthesized water ($Na_2HAsO_4.7H_2O$, 1 ppm)

Kinetic model	**Parameter**	**Value for local well water**	**Value for synthesized water**
pseudo-first	K_1	0.0085	0.0136
	R^2	0.996	0.91
pseudo-second	K_2	0.004	0.003
	R^2	1	0.999
intra particle diffusion	K_{id}	0.214	0.23
	R^2	0.76	0.66
	C	472.76	16.32

4. Conclusions and Recommendations

4.1. Conclusions

In this study, a low-cost adsorbent from the waste material of Corner Brook Pulp and Paper (CBPP) Ltd. was used as a filter media to remove arsenic from the groundwater well. The processed carbon from the ash is found very effective not only to remove arsenic from the arsenic contaminated well waters in the Bell Island, that have the arsenic more than the maximum acceptable concentration but also other elements and components existed in this groundwater. After the cleaning process of CBPP fly ash, two different methods applied for activation: activation with the pure carbon dioxide (CO_2) and, the mixture of steam and CO_2. Both activations significantly increase the surface area and pore volume of carbon sample. The highest surface area and pore volume achieved through the activation with the mixture of CO_2 and steam (CSAC) at 850 ºC for 2 hours, which are much higher than the surface area and pore volume of the cleaned and not activated CBPP fly ash.

While iron impregnation is recommended for increasing the arsenic removal from water, the lowest iron concentrations for impregnation on CSAC are more effective for arsenic adsorption compared to the high iron concentrations, since in the lower concentrations of iron, surface area does not decrease significantly and no pore blockage happens. The impregnated CSAC carbon samples are able to remove the arsenic (V) and arsenic (III) from synthesized water up to 99.63% and 86.64%, respectively. The iron distribution is also found as an important parameter during the impregnation of activated carbon for arsenic removal, since accumulation of iron ions in one spot decrease the surface area and cause pore blockage. The Langmuir model better fits with equilibrium data of arsenic adsorption than the other models. Moreover, the pseudo second order kinetic model is able to explain the kinetic behavior of arsenic sorption for both local well water and synthesized water better than the other models.

4.2. Recommendations

There are some recommendations also about this study that could be done:

i. Multi component adsorption is one of the important issues that should be studied thoroughly, because the existence of some other elements and components affect the arsenic removal efficiency.

ii. Desorption of the adsorbed elements and especially arsenic should be also investigated to find out whether the adsorbent could be reused for treatment or not and also whether it is cost-effective or not.

iii. The economic aspects of this experiment should be examined.

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Appendix A

Figure 0-1: Water well #16 in the Town of Wabana

Figure 0-2: Water well #13 in the Town of Wabana

Figure 0-3: A photo in front of the Wabana town hall