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The Study of Modified Bonechar Synthesis to Apply in the Treating Alum Water in KienGiang Province

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Abstract

In this research, bone char was synthesized going from bovine bone waste which collected at cooking shop of noodle soup with beef in Vietnam country, called as Vietnamese bone char (VNBC). In VNBC synthesis, the results of synthesis process were found the VNBC product sample with the optimal calcination condition to be at temperature of 600 oC, corresponding to the time of 90 minutes. Further, the characteristics of VNBC product sample were analyzed with the specific surface area of 76.404 m2/g, and pore diameter of 87.48 Å measured by BET method. The surface morphology with the particle size measured by SEM method was 70 nm. The composition compounds of VNBC contained calcium carbonate-hydroxyl apatite with the formula was Ca5(PO4)3(CO3)(OH) measured by FT-IR and XRD methods, respectively. In application, VNBC uses as adsorbent to treatment the alum water sample collected at KienLuong region of KienGiang province were considered. The results of VNBC adsorbent to treat alum water solutions measured by UV/VIS method obtained the conditional indicators after alum water adsorption are in agreement with the cleaned water of Vietnamese standard. These results suggested that VNBC are able to use making a potential adsorbent in the treatment of alum water resource becoming clean water resource in Vietnam

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I. INTRODUCTION

The use of meat or fish making food for human has been disseminated in all over the world. This means that the slaughterhouses have to produce an enormous amount of bovine or fish bones. This growing amount of bone residue among other waste materials from meat and fish processing industries considered such as one of the pollutants to environmental challenges that the food industry is facing today. In detail data after slaughtering, one animal is able to produce about 18 weight % bone residues of its total live weight, which considered as slaughterhouse waste source[1-3]. Further, the chemical compositions from bovine bones were consisted with a compound both organic (30%) and inorganic (70%) in which proper mechanical properties of bones as stiffness depended on their mineral parts. The model compound into mineral phase of bones is a biological apatite called as nonstoichiometric hydroxyapatite [4, 5]. This nonstoichiometric hydroxyapatite was used in enamel and dentine with the Ca/P molar ratio exceeds 1.67. Based on its chemical and structural minerals, bovine bone is not only a biocompatible, osteoconductive, non-toxic, noninflammatory and nonimmunogenic agent, but also bioactive to apply greatly in biochemistry[1,5].

In previous bone residues, inefficient management of these bones are creating an effective variety of problems to the endanger public health and the environment in general. When bones are waste in land, this bone wastes created a pollution of surface and underground waters and air quality[6, 7]. As a result, it is caused the affective health of residents

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living within the vicinity of the abattoirs, destroyed water bodies making affectively to fish yield thus [8,9]. In addition, bone wastes are also ideal breeding grounds for disease causing organisms (pathogens) to development. It is usually disposed by burning since treatment, effected thus to which further pollutes the air and endanger human lives.

Adsorption is a phenomena to known that as the consist in the transfer of an adsorbate from a gas or liquid phase to an adsorbent, in which it existed a bonding through intermolecular forces. In adsorption, the quantity of adsorbate in a surface area unit is small. Because of this, normally porous adsorbents with a great internal surface area are selected. On the other hand, it has two types of adsorption phenomena that can be know such as physical and chemical. Physical adsorption, easily reversible, is the result of Van der Waals intermolecular forces (physical interaction) between adsorbent and adsorbed substance (adsorbate). This interactions is weak, thus the energy when the molecule physisorbed is about the same order of magnitude as the condensation enthalpy[10-12]. Whereas, chemical adsorption is the result of the chemical interaction between the adsorbent and the adsorbate to form the chemical bonds. Hereafter, the chemical bond force is able to vary so that chemical compounds cannot be formed in the usual way. However, the adsorption forces of chemisorption is normally a lot higher than of physisorption. During chemisorption, the heat is always released, and as an exothermic process, which commonly is similar to a chemical reaction heat. In addition, it is the same substance that at low temperatures experiences essentially for physical adsorption, sometimes exhibited for chemical adsorption at higher temperatures, and can be both processes simultaneously[13-15]. In application, the adsorption process has a widely application in environmental treatment. Specially, liquid–solid adsorption systems are based on the ability of certain solids as adsorbents to preferentially concentrate specific substances (adsorbates) from solutions onto their surfaces. This rule can be used for the removal of pollutants, such as metal ions and colored organics from wastewater. To adsorbents producing used for adsorption process, a number of low-cost agricultural wastes such as mud tire rubber, and ash are investigated. Other potential minerals and materials for instance bentonite and bone char as potential adsorbents have been tested also, respectively. Many previous studies have been reported during the last ten years to find the low-cost, and efficient adsorbents for the removal of metal ions or color compounds in wastewater [16-20].

Actually, bone char has been successfully synthesized and used for fluoride removal in Tanzania and Thailand [21,22]. The effectiveness of bone char as an adsorbent has been performed using batch experiments with contained heavy metal ions and color solutions under different conditions, for example, variable pH, temperature, chemical and thermal activation, adsorbent dosage [17,18,23]. However, the synthesis process of bone char and its application in wastewater treatment containing metal ions and color solutions at Vietnam (VNBC) are still new research. Bone char is a material obtained by carbonizing animal bones. It is consisted mainly compound of hydroxiapatite and carbonate with its zero charge point between pH value of 8 and 9. Bone char has been used for thousands of years in many fields such as fertilizer, pigment, cosmetics, and varnish. Recent studies have used bone char to adsorb radioisotopes of antimony and europium ions from radioactive wastes. Researchers claimed that sorption is due to cation exchange of metal ions onto hydroxyapatite [14,16,22-25]. Therefore, the goal of this study is to find the process of VNBC synthesis from bovine bone waste. Simultaneously, VNBC product then is measured by analysis methods to determine the surface properties. Finally, we try to apply VNBC product as an adsorbent to remove the organic colors in solution. In the detail this works, the samples of bovine bone waste are treated and calcinated then in conditional without oxygen at

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different temperature and time to find the optimal condition burning. The optimal VNBC sample, then are chosen and measured surface properties by analysis methods. The effects of conditional temperature and time to adsorption capacity on VNBC are also investigated.

KienGiang is a coastal province in the Mekong Delta in southern Vietnam, with most of the province's area in the former RachGia province. KienGiang has a city, a town and 13 districts, a total of 145 communal units including 12 towns, 15 wards and 118 communes. KienGiang has four main land areas: sweet alluvial areas in the west of HauGiang, areas affected by alum inundated in Long Xuyen quadrangle, saline areas in Ca Mau peninsula and mountainous areas, islands in PhuQuoc and KiênHải districts. In particular, agricultural land, accounting for 64.2% of natural area, forest land accounts for 122.8 thousand hectares, special-use land of 35.4 thousand hectares and residential land of 10.1 thousand hectares. In addition, the province has over 70,000 hectares of unused land and unstable production with more than 25 thousand hectares of mixed gardens. Forests in KienGiang are few, mainly protection forests. KienGiang has a monsoon tropical climate, hot and humid all year round. KienGiang is not directly affected by the storm but the amount of rain due to storms accounts for a significant proportion, especially at the end of the rainy season. Weather conditions of KienGiang have the basic advantages that other provinces in the Mekong Delta do not have such as: lost natural disasters, no cold, no direct landing storms, abundant light and stable temperature are during the year. It is very convenient for many kinds of plants and animals to grow. In KienGiang province, the level of acidity is quite high, ie low pH, people call it alum water because of the sour taste. Acid in alum water is sulfuric acid, which is formed when alkaline pyrite (FeS_2) soil comes into contact with air. Acid sulfate soil was formed due to geological tectonic process. In the rainy season, rainwater washes out acid sulfate soils, carries a lot of iron, aluminum sulfate and organic humus acid, contains lots of H ions and acidic hydrolysates such as $AlCl_3$, $Al_2(SO_4)_3$, $FeCl_3$, $Fe_2(SO_4)_3$, $FeSO_4$. Through the pollution of acid sulphate soil, most of the regions from Ha Tien to alleviate alum water affect the living process of the people here, especially the consequences of alum water cause such as causing stomach pain, water containers are corroded, bathed with blisters, opaque yellow water containing a lot of iron alum causes poor aesthetic feeling, clear blue water contains a lot of alum, low pH, if Use will damage tooth enamel, digestive system because water is too acidic. Iron content greater than 0.3 mg/ L, manganese greater than 0.1 mg/ L stains clothes and household containers, high aluminum content will make water colored and deposition in containers, causing neurological disorders, causing osteoporosis in the elderly and affecting the kidney's ability to filter blood; high sulfate content causes unpleasant taste for broth. The data show that the composition and image of alum water collected in KienGiang province shows the time according to the table 2 below.

Base on current situation of the bovine bond waste and alum water sources at KienGiang province of Viet Nam that need to be solved. Therefore, the important applications of bone char can be seen that the construction of bone char synthesis process and its application in treating alum water are necessary to deploy. Thus, studies need to be developed, experimented, as well as knowledge to run on a small scale first needs to be done. To do this, the first is to build the process of synthesizing bone char and its product application then in environmental treatment of alum water with the scale of applied research in the laboratory, this is the main works in the project, which we did.

II.MATERIAL AND METHODS

Many samples of the bovine bone wastes collected from cooking shop of noodle soup with beef, and experiments to VNBC synthesis are performed at the

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Faculty of chemical engineering, Industrial University of Ho Chi Minh City of Vietnam. In all experiments and measurements are that the first step, we fixed each time (time constant) to calcine the bovine bond samples to VNBC products with the changed temperature from 500-800 0C, and trying to then adsorption capacity of VNBC products with alum water. Hereafter, the highest adsorption capacity product is found to correspond with optimal temperature burning. In second step, by repeating similar experiments in the first step, temperature (temperature constant) is fixed but to find optimal time burning. Finally, VNBC product in synthesis process having an optimal temperature and time burnings were found, and measured the surface characteristic by analysis methods.

Samples of alum water were collected in the district of KienLuong district, the pH was 2.5. The samples were taken at the last March 2019, analyzed then to determine the chemical composition of initial alum water sample which it listed in Table 1.

Table 1. Data for chemical composition of alum water before treatment

No.	Indicators	Number of samples analyzed	Concentration (mg /L)
1	Độ pH	3	5.2
2	Fe^{3+}	3	4.0
3	Al^{3+}	3	1.3
4	Pb^{2+}	3	0.05

In performance of alum water treatment by VNBC products, the concentrated alum water after adsorption on VNBC was analyzed by UV-VIS spectrophotometer. Herein, this method consists in forming alum water complex which are measured with photometric measurement of absorbance at a wavelength of 550 nm and 650 nm, respectively.

To determine the surface characteristics of optimal VNBC product such as compound components and phase composition are analyzed by X-ray Diffraction (XRD) method with graphite monochromator using Cu-Kα of 1.54, combined with FT-IR analysis spectrophotometer in the range of middle infrared from 400 to 4000 cm–1. In addition, the effect of calcination temperature on the microstructure of optimal VNBC sample is analyzed also using scanning electron microscope (SEM). The calculations of adsorption capacity analysis methods are used in this work to be in line with previous experiments[26, 27]. Many results of synthesis process and analysis are performed respectively in following below,

III. RESULTS AND DISCUSSION

III.1.The process to VNBC synthesis

The samples of bovine bone waste are collected from cooking shop of noodle soup with beef, washed with hot water at temperature (T) from 50 to 60 oC, done then the fat clean with weak soap solution. The samples are washed continuously with hot water at 50-60 oC, and dried at 105 oC. Finishing wash, bovine bone wastes are calcinated into furnace to VNBC product, and grinded then to the small pieces of 1 mm particle sizes. The process of VNBC synthesis is at Figure 1 in following:

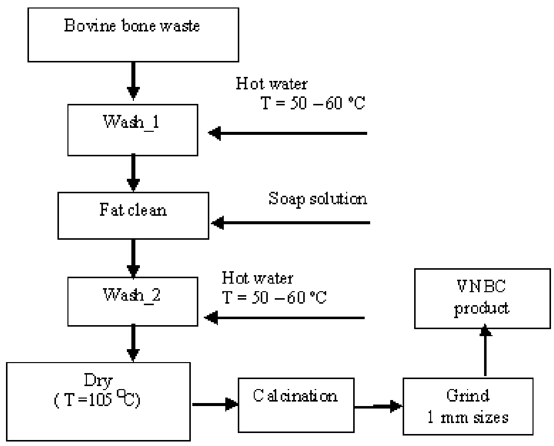

Figure 1: VNBC synthesis process

The most important step of synthesis is bone calcinated into furnace at the condition of without oxygen. The principle and structure of calcinated furnace with laboratory model in charring of bone is designed similarly to previous study[28]. By doing this, the performance of synthesis steps from bovine bone waste to VNBC product was obtained experiments at laboratory as shown in Figure 2.

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Figure 2: The images of VNBC synthesis process in laboratory. It is denoted that Figure (a) as bovine bones collected from cow slaughterhouse, Figure (b) as bovine bones collected from cooking shop of noodle soup with beef, and Figure (c) as bovine bones synthesized to VNBC, respectively.

III.2.The experiments on burned temperature effects to BC synthesis

In this works, we have done experiment that, each a bovine bone samples (BBs) after cleaning is weighed with initial m1 mass (in gram, g), and then burned into furnace at conditional without oxygen at 60 minutes, where it is changed the temperature burning (Tb) from 400 to 800 oC. After burning, VNBC product is weighed to determine the m2 mass (g) in each BBs. The results of burned temperature effect and recover efficiency as the ratio of m2 divided m1, namely E (in percentage, %) of VNBC products at different temperature are obtained in Table 2, respectively.

Table 2.The effects of burning temperature to VNBC synthesis.

BBs Sample	T_b	m_1	m_2	E
500- 60	500	11.16	8.12	72.76
600- 60	600	10.04	6.95	69.22
700- 60	700	10.15	7.01	69.06
800- 60	800	10.56	7.30	69.13

We observed in Table 2 that E is 69.22 % at Tb of 600 oC. Thus, in this works, use of the BB600-60 sample is chosen as VNBC product at optimal condition of 600 oC temperature in synthesis process. Further, VNBC products are charred bone does not add color, taste or smell to the water which it is in line with bone char products in previous experiments[15, 22, 23, 28, 29]. This is able to help us have a confident method in VNBC synthesis, also potential applications of product in next time research.

III.3. The experiments on burned time effects to BC synthesis

In similarly, each a bovine bone samples (BBs) after cleaning is weighed with initial m1, then calcinated at conditional without oxygen at the temperature of 600 oC, whereas it is changed the times burning (Timeb) from 60 to 180 minutes. Hereafter, VNBC product is weighed to determine the m2. The results of burned time effect of VNBC products at different temperature presented in Table 3, respectively.

Table 3.The effects of burning time to VNBC synthesis.

BBs Sample	Timeb	m_1	m_2	E
600- 60	60	19.50	14.70	75.38
600- 90	90	18.94	14.27	75.34
600- 120	120	23.32	17.25	73.97
600- 150	150	23.09	16.74	72.50

It is an observation from Table 3 that the recover efficiency of 75.34 % at time of 90 minute to choose as optimal sample with time. Because, the results of conditional calcination depending on recover efficiency follow us to conclude that the sample of BB600-90 is the most optimal one. This is to say that synthesis process to VNBC product at optimal condition of temperature as 600 oC, corresponding with the time of 90 minute. Thus, we are used then this sample to treat alum water and measure the surface characteristics of VNBC product at conditional optimization in next step, in following.

III.4. Adsorption capacity of VNBC in alum water treatment

In alum water treatment, in each experiment, a ratio of experimental sample of weighed (gram,g) VNBC product and alum water volume (in ml) contained the chemical composition above (see in Table 1) as 1:10 is mixed together. Sample is shaken then at 60 minute with 250 revs per minute, which the time of

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sample changed from 30, 60, 90, 120, and 150 minutes, respectively. After those time, samples are filtrated to obtain alum water after asorption by VNBC, measured then pH values and chemical composition by UV-VIS method to determine concentration (in ppm) of chemical compositon in alum water after adsorption. In addition, we also present the concentration of chemical composition in clean water standar in Viet nam. The resulted experiments of five samples (sample 1-5) are found and listed in Table 4, as follow.

Table 4. Chemical composition of alum water after treatment by VNBC product

No.	Indicators	Standard of clean water in Vietnam (ppm)	Concentration after adsorption					
			Initial sample	Sample 1	Sample 2	Sample3	Sample 4	Sample 5
1	pH	6,0-8,5	5.2	6.5	6.25	6,8	6.7	6.8
2	Fe^{3+}	5.0	4.0	3.3	3.2	3.7	3.8	3.9
3	Al^{3+}	2.5	1.3	1.2	1.1	1.2	1.2	1.1
4	Pb^{2+}	0.5	0.05	0.0 3	0.01	0.04	0.02	0,02

It is observed the chemical concentration after adsorption of alum water sample in Table 4 that the increased pH value is reasonable to compare with sample of clean water standard. To explain for this increase of pH value is clearly that hydroxyl group in the structural VNBC neutralized with H+ in alum water, this cause makes pH increase. Further, all concentration ions in alum water such as Fe+3, Al+3, and Pb+2 is more decreased respectively after adsorption in comparison with clean water sample. Simultaneously, at the time of 60 minute on sample of 2, the adsorption capacity of VNBC is the most one because the concentration after adsorption as being lowest, which it followed us to conclude that adsorption time of alum water is the optimal condition of 60 minute. In addition, these results are to say that VNBC being a strong adsorbent with heavy metal ion in alum water. Therefore, our resulted study is giving a positive in alum water treatment by synthesized VNBC product, which can be applied in industry.

III.6. The resulted characteristic surface of optimal VNBC

III.6.1. BET analysis

This analyzed techniques were based on the combination between theoretical BET [31,32] to determine the pore diameter and specific surface area of adsorbents exhibiting a wide range of pore sizes, and described appears to be applicable to porous solids of any nature. By this method thus, the optimal VNBC sample is plotted contour by BET method to what measured results, presenting in Figure 3.

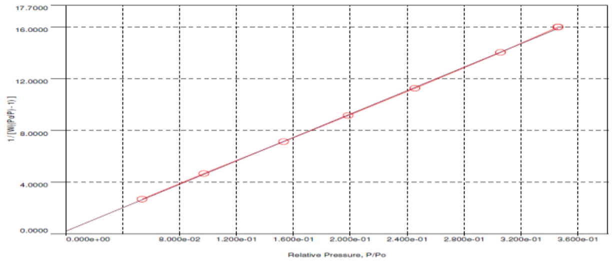

Figure 3. Chart of BET results of VNBC calcined at 600 oC for 90 minutes

The resulted BET of VNBC calcined at 600 oC for 90 minutes, the graph can give the following,

Slope = 45.358

Correlation coefficient R = 0,999920

C constant = 204.803

Surface area = 76,404 m²/g

The measured results is observed from Figure 3 that optimal VNBC sample has a specific surface area to be 76.404 m2/g with pore diameter of 87.48 Å (8.75 nm), this is able to say that VNBC such as mesoporous material has a good adsorption capacity to which it has many potential applications in industry. The resulted BET analysis of specific surface area in present work is varying interval with BET analysis in previous investtigation [27], this may be due to the differnce of method or different animal bone which effect to synthesis process.

III.6.2. SEM analysis

SEM analysis is confirmed that VNBC product at optimal condition to demonstrate a diversified

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propensity for crystal agglomeration on the surface. Inhere, the surface morphology and crystal size of optimal VNBC sample (BB600-90) is measured by SEM method, shown in Figure 4.

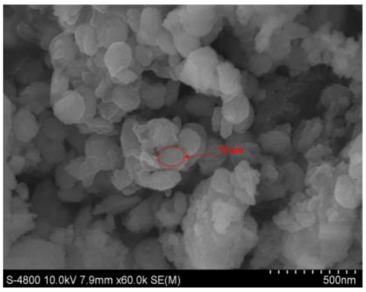

Figure 4. Resulted SEM of optimal VNBC sample with the scale of image is noted 500 nm

It is an observation at Figure 4 that the microcrystal of VNBC sample contributed to be uniform and small particle size. To be more clearly, a scale of VNBC at 500 nm is taken with image to give a crystalline size is 70 nm. Resulted SEM image in this study is in line with previous experiment [27], this means that VNBC synthesis method has a suitable.

III.6.1. FTIR analysis

The basic of FTIR analysis is to identify the characteristic of functional groups on adsorbent surface such as VNBC in present work. The resulted FTIR spectra is measured, and shown in Figure 5.

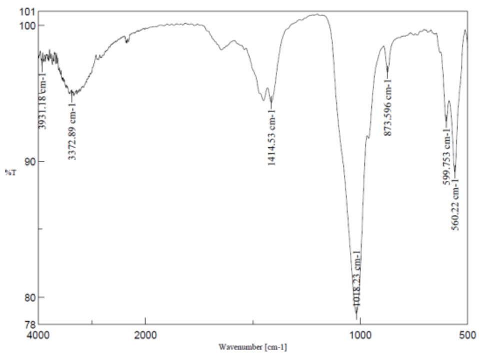

Figure 5. FTIR results of bovine bone after calcined at optimal condition of BB600-90 sample

FTIR analysis of bone char products from animal bones in previous experiment [27,30,33] have been reported that, in transmittance mode, shown the presence of carbonate group at around 1410-1450 cm^{-1} and 873 cm^{-1}, carbon group at C-C (1200 cm^{-1}), C=C (1650 cm^{-1}), and C≡C (2150 cm^{-1}), with hydroxide group at around 3500-3200 cm^{-1}, respectively. For phosphate group at 1030-1090 cm^{-1} and 1950-2200 cm^{-1}, 962 cm-1 and 560 cm^{-1}. Herein, we observe FT-IR at figure 5 that the bands corresponding to the vibrations of C–H and C–C bonds of organic compounds is absent which this confirmed that a total organic matter was removed after the synthesis process. It is only weak band of C=C bond at 1652 cm^{-1} which we predicted that the VNBC sample existing with carbon black composition. Continuously, the presence of characteristic bands of phosphate group (PO_4^{3-}) is resulted that the sharp peaks existing the wave number of 560 cm^{-1} assigned to bending mode of phosphate and 599 cm^{-1} for the presence of hydroxyl group (OH). The most intensive bands of 1018 cm^{-1} is corresponded to stretching vibrations of P–O bonds, whereas the wave numbers of 3372 cm^{-1} came from the symmetric stretching vibration of H–O bonds. In addition, the low-intensity bands at 1414 and 873 cm^{-1} is observed for sample corresponding to the vibration mode of carbonate group (CO_3^{-2}). These follow us to conclude that the optimal VNBC sample is contained the structure of carbonate hydroxyl apatite compounds.

III.6.4. XRD analysis

To determine the phase composition, the optimal VNBC sample is measured using XRD analysis. The analyzed results of VNBC sample at optimal condition presented in Figure 6.

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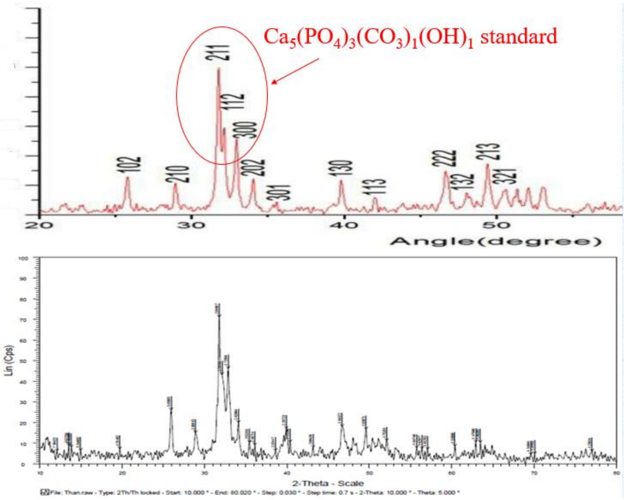

Figure 6. The resulted spectrum of optimal VNBC sample measured by XRD of bonechar samples burned at 600°C for 90 minutes

We observed the XRD spectrum in Figure 6 that, the peaks of VNBC sample (black solid line) is overlapped with the peaks of standard $Ca_5(PO_4)_3(CO_3)_1(OH)_1$ sample (red solid line) at diffraction angle (2θ) from 30 to 35^0, this confirmed that VNBC sample existing the composition of $Ca_5(PO_4)_3(CO_3)_1(OH)_1$ compound(calcium carbonate-hydroxyl apatite. The XRD analysis in this work is an agreement with previous studied [15,27,29,30] to which confirm again that the method of VNBC synthesis process is successful.

IV. CONCLUSIONS

From the resulted investigations, we draw some conclusions in following,

1. 1. In VNBC synthesis, many samples of bovine bone are performed in laboratory which optimal VNBC product sample found at Temperature of 600 °C, corresponding with the time of 90 minutes.
2. The use of VNBC product as an adsorbent is examined to treat alum water in KienGiang province. The results of VNBC adsorbent to remove heavy metal in alum water is found and obtained with five experimental samples after adsorption which is reasonable with the Vietnamese standard of clean water sample. This is required that used VNBC as adsorbent to treat alum water resource in Vietnam.
3. Many analyses on optimal VNBC sample were measured to determine the surface characteristics. In these analysis methods, SEM can be helpful to describe the morphology of VNBC at nanoscale level with a crystalline size of 70 nm. FTIR and XRD technique given us to find out that crystalline nature of VNBC, existing the calcium metal is bound with PO_4^{-3}, CO_3^{2-}, and OH^- groups, to which structural crystal was $Ca_5(PO_4)_3(CO_3)(OH)$ compound (calcium carboxyl-apatite). Furthermore, BET technique can be used to distinguish the types of pore material and adsorption capacity of adsorbents. Hence, the resultedspecific surface area of 76.404 m^2/g with pore diameter of 87.48 Å (8.75 nm) measured to what VNBC sample in this study is a good adsorbent and as a mesoporous material having potential applications in industry.

This study is the first step to design successfully the process of VNBC synthesis, also apply it to be an adsorbent in alum water solution treatments. Many other applications and kinetic model of VNBC will be studied continuously in next research.

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