Received: 6 September 2019 | Accepted: 9 December 2019

DOI: 10.1002/ces2.10034

ORIGINAL ARTICLE

International Journal of Ceramic Engineering & Science

Changes in fluoride removal ability of chicken bone char with changes in calcination time

Masanori Kikuchi[1] | **Yuki Arioka**[2] | **Masamoto Tafu**[3] | **Mitsuteru Irie**[4]

[1]Bioceramics Group, National Institute for Materials Science, Tsukuba, Japan

[2]Master's Program in Life Science Innovation, University of Tsukuba, Tsukuba, Japan

[3]Department of Applied Chemistry and Chemical Engineering, Toyama College, National Institute of Technology, Toyama, Japan

[4]Faculty of Engineering, University of Miyazaki, Miyazaki, Japan

Correspondence
Masanori Kikuchi, Bioceramics Group, National Institute for Materials Science, 1-1, Namiki, Tsukuba, Ibaraki, Japan.
Email: kikuchi.masanori@nims.go.jp

Funding information
Japan Society for the Promotion of Science, Grant/Award Number: 25257306

Abstract

Changes in fluoride removal ability of chicken bone char (CBC) were investigated by both remained amounts of carbon including organic substances and crystallite size of hydroxyapatite in the CBC. Carbon contents in CBC were controlled by heating time at 600°C. Although temperature for crystal-grain growth for HAp, 650°C, was higher than 600°C, crystallite size of HAp in CBC increased with heating time. Fluoride ion removal ability positively related to the amount of remaining carbon and negatively related to the square of crystallite size, as an index of surface area, of HAp. These results suggested that fluoride ion removal from water by CBC is not only by ion exchange and/or dissolution-precipitation process, but also by adsorption by carbon and/or temporal capture of fluoride ion by microstructure of carbonate in CBC before immobilize it in apatite structure.

KEYWORDS

chicken bone char, calcination time, carbon content, crystallinity, fluoride removal

1 | INTRODUCTION

Groundwater in Middle East and North Africa (MENA) area is an irreplaceable water resource because of its accessibility and future sustainability of water quantity.[1,2] Further, natural contamination of drinking water such as groundwater with various toxic ions has been reported in many regions in the world, and high concentration of these toxic ions such as heavy metals, fluoride, and arsenic in the groundwater raises a risk level for human health.[3,4] Focusing on fluoride ion, its excess intake causes a serious problem on teeth and bones that called dental or skeletal fluorosis.[5] For example, huge numbers of dental fluorosis had reported in Kairouan, Tunisia, due to high concentration of fluoride in the groundwater around there.[6] As the same situation as in Tunisia, serious dental and skeletal fluorosis patients found in Turkey had taken groundwater containing high concentration fluoride ion as a drinking water.[7] Reverse osmosis and coagulation-precipitation with activated alumina[8] are widely applying to removal of fluoride ion from drinking water. These methods remove fluoride ion from the water to satisfy the WHO drinking water standard, <1.5 mg/L; however, materials and reagents for these removal methods are expensive for developing countries.[9] Accordingly, those conventional methods can apply to drinking water insufficiently in developing countries. Therefore, low-cost fluoride removal method is strongly desired.

Fluoride ion removal properties of bone chars (BC), obtained by calcination of waste bones from edible animals, cow,[10] pig,[11] and chicken,[12,13] have been studied. Chicken bone char (CBC) is expected as an ideal material

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for fluoride ion removal agent because chicken bone can be obtained at low cost as a waste from poultry and is free from religious taboo. Bone char consists of major two components: One is nonstoichiometric carbonate containing hydroxyapatite (HAp, $Ca_{10}(PO_4)_6(OH)_2$),[14] the main inorganic component of bone, and the other is carbon from charring of organic substances of bone. Two F^- ion removal mechanisms of HAp have been proposed: One is ion exchange with OH ions in HAp, and the other is a dissolution-reprecipitation process of HAp with a substitution of a part of OH^- ions with fluoride ions.[15] The latter process would be accelerated for CBC due to higher solubility of nonstoichiometric HAp in CBC than stoichiometric HAp.[16] Influence of carbon in BC on fluoride removal was investigated and by Larsen et al. They concluded that carbon was an inhibitor of fluoride ion removal without showing the concrete evidence in their paper.[17] Contrarily, carbon, prepared from waste carbon slurry[18] and charfine,[19] showed removal of fluoride ion from water efficiently. Thus, carbon in BC would have possibility to contribute to fluoride ion removal.

Terasaka et al[11] and Larsen et al[17] investigated fluoride ion removal properties of BC prepared by heating at various temperature and reported that changes in pyrolysis temperature of CBC affect to the crystallinity of HAp in CBC. Motoyama et al reported that the difference of pyrolysis temperature induced changes in the crystallinity of HAp in chicken bone and affected to fluoride ion removal property.[13] Therefore, carbon content control with maintenance of crystallinity of HAp in CBC is necessary to consider the influence of carbon in CBC on fluoride ion removal property. The amount of carbon in ox BC could be controlled by changing pyrolysis time of bone char.[17]

In this study, CBCs with different carbon amount were prepared by calcination of raw chicken bone for different time. The CBCs obtained were characterized their carbon contents, crystal phases, crystallinity, and fluoride ion removal abilities.

2 | MATERIALS AND METHODS

2.1 | Materials

Waste chicken humeri were used as a raw material of CBC. The humeri were pretreated by boiling 1 hour with distilled water and drying 24 hours at 105°C in order to remove remaining fat and meat residue. The preprepared humeri (raw chicken bone, RCB) were heated with a programmable electric furnace (AS ONE, MMF-1) in air at 600°C, which is the sufficient temperature to pyrosis of organic substances in bone[17] as well as lower temperature than that, 650°C, of HAp crystal-grain growth,[20] for 0.5, 1, 3, 6, 12, and 24 hours at a heating ratio of 10°C/min. The CBCs obtained and an evaporating basin were quickly removed from the furnace and then cooled at room temperature in air. The CBCs obtained were crashed, washed with distilled water, and separated particle by sieve that opening was 150 μm. A sodium fluoride standard solution (Wako Pure Chemical Industries, Ltd.) was used for preparation of starting fluoride ion solution for fluoride removal ability test of the CBCs by diluting with pure water.

2.2 | Characterization of raw chicken bone and chicken bone chars

Thermal changes of the RCB and CBCs up to 1000°C were analyzed by a thermogravimetry-differential thermal analysis (TG-DTA, Thermo plus EVO TG8120, Rigaku, Co. Ltd.) using platinum pan as a sample container and fine alumina powder as a reference material at a heating rate of 10 K/min. Amounts of organic substances in the RCB and carbon in CBCs were calculated from the weight loss of TG. Crystal phases of the RCB and CBCs were identified by a powder X-ray diffractometry (XRD, MiniFlex, Rigaku, Co. Ltd) using Cu$K\alpha$ radiation at a scanning rate of 2°/min in 2θ.

Full width at half maximum (FWHM) of HAp 002 diffraction for each specimen was collected with a RINT200 (Rigaku, Co. Ltd) using graphite monochromatized Cu$K\alpha$ radiation by step scan at a step width of 0.01° and collection time of 1 second. Crystallite sizes for *c*-axis of HAp in CBCs were calculated from the FEHM by Scherrer's equation using 800°C-burnt synthesized HAp as a sufficient high crystalline HAp for removing of diffraction width from the XRD apparatus.

2.3 | Evaluation of fluoride removal ability of chicken bone chars

2.3.1 | Fluoride removal by chicken bone chars as a function of time by a jar test

Fluoride ion removal by the CBC as a function of time was measured by a jar test. Experimental apparatus is illustrated in Figure 1. Three-hundred milliliters of starting fluoride ion solution at 10 mg/L was added into the cylindrical reaction flask with flat plate baffles (3026-05/85A-A, Asahi Glassplant Inc.) with a maintenance of solution temperature at 25°C with the water bath (HB10, IKA). One gram of the CBC was then added to the solution and stirred at 200 rpm with the paddle type impeller with shaft (Stainless paddle shaft, Toyama Sangyo Co., Ltd.) driven with the motor (FLOS20-S, AS ONE). Fluoride ion concentration

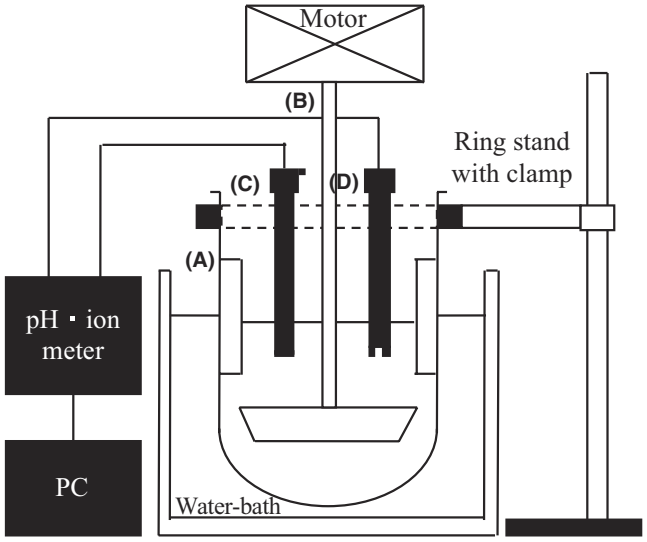

FIGURE 1 Schematic drawing of experimental equipment for jar test. A, Cylindrical reaction flask with flat plate baffles; B, paddle type impeller with shaft; C, fluoride ion selective electrode; D, pH electrode

and pH were continuously collected with PC and software (LabX direct pH, Mettler Toledo) using the pH/ion meter (SevenExcellence S500, Mettler Toledo) equipped with the fluoride ion selective electrode (Orion 9609BNWP, Thermo Fisher Scientific Inc.) and pH electrode (InLab Expert Pro ISM, Mettler Toledo) up to 100 hours.

2.3.2 | Fluoride removal by chicken bone chars at 40 hours by a shaking test

A fluoride removal test by the CBC at 40 hours was measured by a shaking test. The CBC of 50 mg was added into 20 mg/L of 20 mL fluoride ion solution in 50-mL centrifuge tube. The mixture was then shaken for 40 hours with a reciprocal shaker (NR-1, TAITEC Corporation) at 120 rpm and room temperature. After the shaking, the mixture was pressure filtered with a 0.45-μm syringe filter, and fluoride concentration of the filtrate was measured with the pH/ion meter equipped with the fluoride ion selective electrode. A fluoride removal capacity was calculated according to the following equation modified from Dahi et al[21] for a shaking time, from 15 to 40 hours, and correction for net amount of bone minerals using carbon content calculated from the TG-DTA data;

$$F_{\mathrm{CBC}} = (C_0 - C_t)\frac{M_{\mathrm{CBC}}}{V_{\mathrm{F}} \times R_{\mathrm{HAp}}}$$

where F_{CBC}, fluoride removal ability of the CBC (mg/g); C_0, initial fluoride concentration (mg/L); C_t, fluoride concentration after 40 hours treatment with the CBC (mg/L); M_{CBC}, mass of the CBC added (g); V_{F}, volume of the fluoride solution (L); R_{Hap}, ratio of HAp in the CBC.

3 | RESULTS AND DISCUSSION

3.1 | Thermal analysis of raw chicken bone

Figure 2 shows the result of TG-DTA of the RCB. The early weight loss observed in the range of 30-150°C with the endothermic reaction was a removal of H_2O in the RCB.[22,23] The huge mass loss with high exothermic reaction in the range of 250-350°C was the combustion of organic substances in the RCB mainly collagen.[22] The continuous weight loss had continued from 350 to 550°C with an exothermic reaction could be combustion of other organic substances with higher pyrosis temperature in bone matrix. These results agreed with previous reports, and calcination temperature, 600°C, for the RCB to prepare the CBC was sufficient temperature.

3.2 | Colors and carbon amounts of chicken bone chars

Figure 3 shows the photograph of CBCs calcined for or different times 0.5, 1, 3, 6, 12, and 24 hours. Colors of CBCs were changed in different calcination time from dark gray to white. Sample name, color, and mass loss of the RCB and CBCs are summarized in Table 1. White substance in CBC is bone mineral, apatite, and black substance in that is considered as charred organic substances in the RCB, *that is*, carbon. Therefore, darkness of CBC could depend on a mixing ratio of apatite and carbon in CBC.

Carbon amounts of CBCs were calculated from the mass loss in the TG curves ranging from 200 to 800°C according to the Terasaka's method.[11] Figure 4 shows the relation between carbon amounts and calcination times of CBCs.

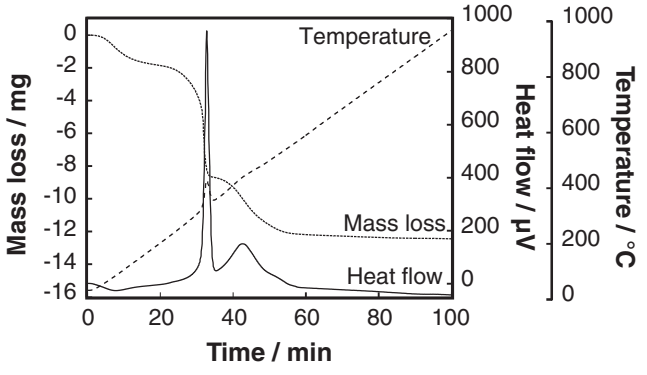

FIGURE 2 Thermogravimetry-differential thermal analysis curves for raw chicken bone

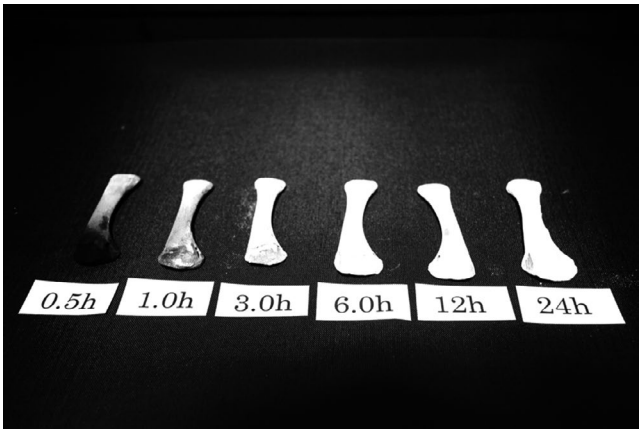

FIGURE 3 Chicken bones calcined at 600°C for 0.5, 1, 3, 6, 12, and 24 h

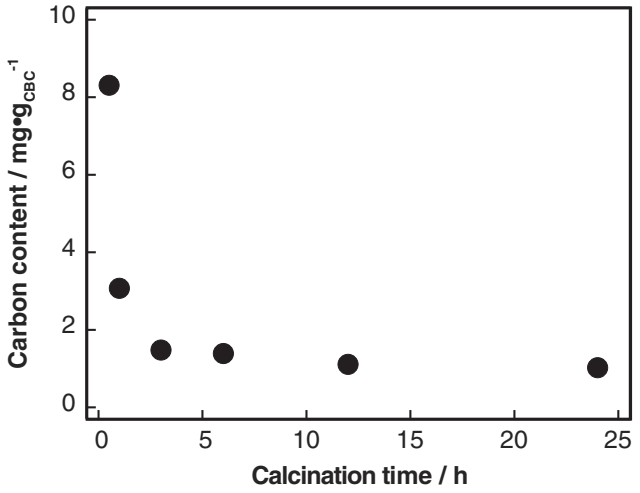

FIGURE 4 Carbon contents in chicken bone chars as a function of calcination time

The CBC0.5, calcined for the shortest time in the present study, exhibited the highest carbon amount. The CBC6, CBC12, and CBC24 have an approximately 1 mg carbon in 1 g of CBC and showed no significant differences in carbon amounts. These results suggested that carbon amount in CBC was easily controlled by calcination time in air at 600°C.

3.3 | Powder X-ray diffraction analysis on raw chicken bone and chicken bone chars

Figure 5 shows XRD patterns of RCB, CBCs, and synthetic HAp (Food grade, Taihei Chemical Co., Ltd.). All peaks of RCB, CBCs, and synthetic HAp were ascribed to HAp (JCPDS 9-432), and no other crystalline phases were detected. The XRD pattern of RCB showed broad peaks due to low crystallinity of HAp in RCB. Peak widths decreased with increasing in calcination time. Three strongest peaks of HAp were completely merged in one peak in RCB and CBC0.5, however, barely identified in CBC24. Crystallite sizes of RCB and CBCs calculated from 002 are summarized in Table 2. These crystallite sizes calculated from Scherrer's equation theoretically contain also crystal distortion of HAp crystals in RCB and CBCs. Therefore, these results suggest that crystal growth including ion re-alignment occurred even below the generally considered "crystal-grain growth" temperature. In HAp crystal formation in water at low temperature up to body temperature, hydrated layer on HAp nanocrystals plays a role for the crystallization of HAp nanocrystals by acting as a sort of "vehicle" for movement of ions and ionic groups in HAp nanocrystals to stabilize conformation of ions in the crystal structure of HAp.[24] In addition, water

TABLE 1 Color of raw chicken bone and chicken bone chars after calcination at 600°C for different calcination time

Sample name	Calcination time/h	Color after calcination
RCB (no heating)	—	Tarnish
CBC0.5	0.5	Dark gray
CBC1	1	Light gray
CBC3	3	Off white
CBC6	6	White
CBC12	12	White
CBC24	24	White

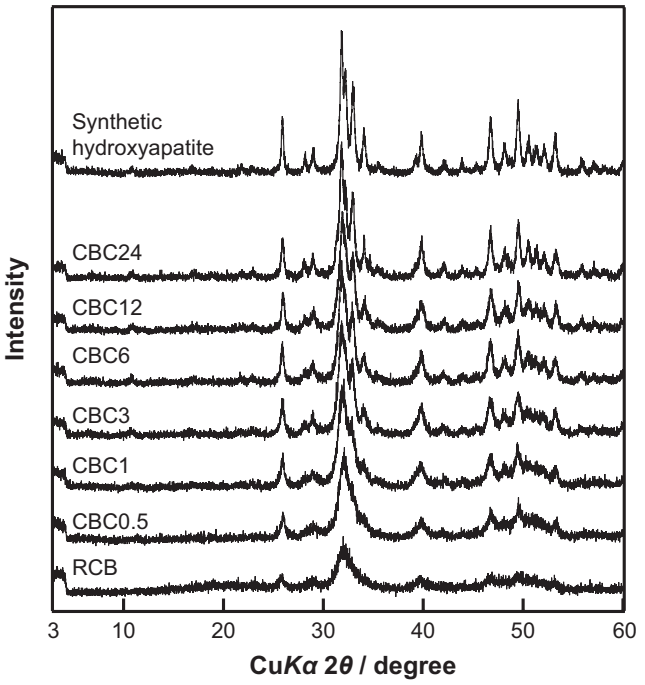

FIGURE 5 Powder X-ray diffraction patterns of raw chicken bone and chicken bone chars calcined at 600°C for 0.5, 1, 3, 6, 12, and 24 h

TABLE 2 Crystallite sizes of apatite in CBC after calcination for each time. Square of crystallite sizes is also shown as a function of surface areas of apatite crystals in CBCs

Sample	Crystallite size/10^{-1} nm	Square of crystallite size/10^{3} nm^2
RCB	2.9	0.84
CBC0.5	3.4	1.2
CBC1	3.6	1.3
CBC3	3.9	1.5
CBC6	4.3	1.8
CBC12	6.1	3.7
CBC24	7.7	5.9

including hydroxyl group in HAp is generally interminably removed by heating. This phenomenon demonstrates possibility of the existence of a kind of "hydrated layer" on HAp nanocrystals that have large specific surface area. Further, higher temperature increases ion activities. These two phenomena may be the reason of crystallization of HAp nanocrystals below the crystal-grain growth temperature.

As mentioned above, fluoride removal mechanism of bone char had been reported that HAp in bone matrix is the main contributor of the reaction. Rojas-Mayorga et al[10] confirmed organic substance amounts of bovine bone chars depended on heating temperatures by Fourier-transformed infrared spectroscopy. Terasaka et al[11] revealed that changes of the calcination temperature of bone char affect the removal ability of fluoride ion due to increasing crystallinity of HAp with increasing in calcination temperature. Thus, CBC preparation with changing of the calcination time instead of the changing of the heating temperature was considered to be control the organic matter amount in CBC samples with maintaining of the crystallinity of CBC samples; however, calcination below the generally believed temperature of HAp crystal-grain growth was not sufficient to inhibit maintaining of the crystallinity as shown above. Therefore, the following F^- removal was considered with both carbon contents and crystallite sizes as a function of crystallinity.

3.4 | Fluoride removal ability of chicken bone chars by jar test

To assess the fluoride removal ability and treatment speed of CBC samples, jar test was performed using the experimental equipment (Figure 1) and two types of CBC samples: CBC0.5 and CBC24. Figure 6A shows the results of the fluoride treatment by using CBC0.5 and CBC24. Vertical axis shows residual concentration of fluoride in the solution, and horizontal axis shows treatment time. In the case of CBC0.5, fluoride concentration sharply decline within a few hours and resulted at 18% by 40-hour treatment. In contrast, the decline of fluoride concentration stopped at 6 hours with respect to CBC that heated for 24 hours, whereas it showed sharply decline to 70%.

From the result in Figure 4, CBC0.5 contained larger amount of organic matter than CBC24; hence, HAp amount of CBC was less than CBC24 in an equivalent amount of CBC samples. This means that much higher removal rate was shown in the case of CBC0.5 in comparison to that of CBC24, if they were normalized by HAp amount, generally believed as playing a role of fluoride ion removal. If the removal mechanism of bone char was only by HAp as reported previously, F^- removal ability of CBC24 can be higher than CBC0.5; however, opposite results were demonstrated. Therefore, carbon in the CBC can support for or contribute to fluoride removal by HAp in it.

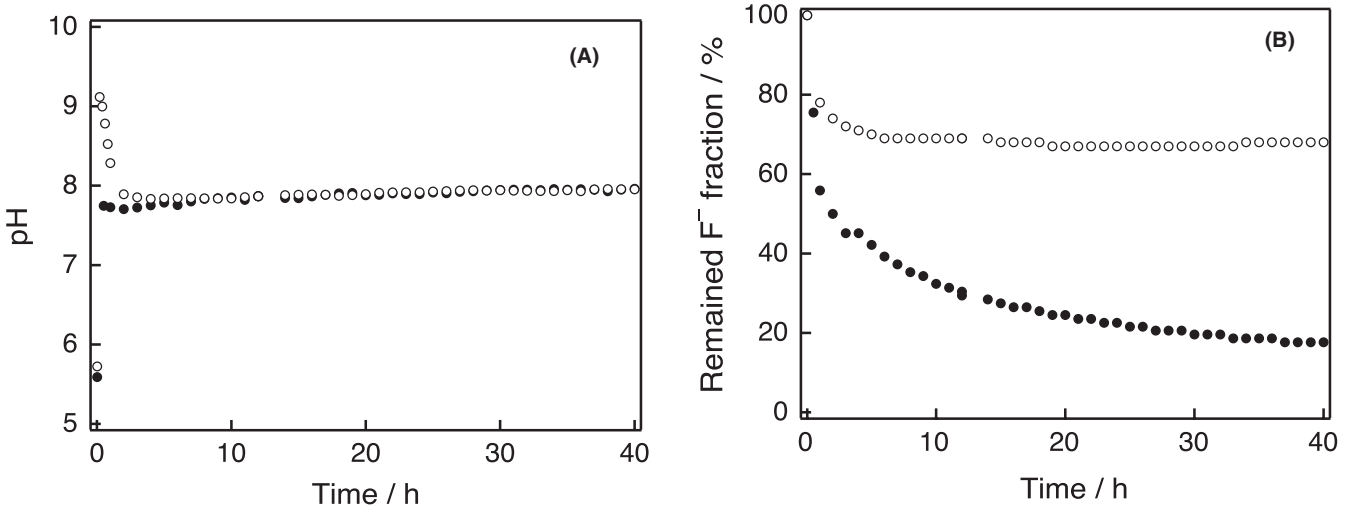

FIGURE 6 Influences of calcination time of chicken bone char for (B) the fluoride removal ability and (A) pH. Closed circles illustrate CBC0.5 and open circles, CBC24

Figure 6B shows the solution pH of each experimental condition. By adding CBC samples, pH of the solution increased in both conditions: CBC0.5 and CBC24. Especially on CBC24, pH value changed drastically within a few minutes. Consequently, the pH values on both CBC samples converged around pH 8. Sbczak-Kupiec and Wzorek revealed that the suspensions of natural hydroxyapatite samples that were obtained by calcination of pig bone showed high pH value around ten to eleven. Additionally, the presence of free carbon oxide had confirmed from the samples.[25] It also indicates free carbon oxide increased the solution pH because it reacts with the water that forming calcium hydroxide and it could be occurred the reaction in these experiments. Afterward, pH value on CBC24 sharply decreased immediately and it seems to be occurred by the dissolution of atmospheric carbon dioxide due to the stirring of the solutions.

According to a previous report, maximum fluoride removal capacity of bovine bone char that was heated in N_2 gas condition showed around 6 mg/g with neutral pH.[10] Furthermore, Medellin-Castillo et al[26] revealed the relationship between fluoride removal capacity of bone char and final pH of the solution. In the case of the final solution pH at 8, the fluoride removal capacity resulted approximately 3 mg/g. This result is in accord with our result of CBC0.5 in Figure 6 (data not shown). In addition, as mentioned above, the presence of free carbon oxide on CBC could affect the solution pH. Thereby, it could be considered that the early decline of fluoride concentration in the case of CBC24 was attributed by the increase of the pH that caused by the forming of calcium hydroxide. Finally, fluoride concentration on CBC0.5 continuously decreased even after CBC24 became equilibrium. Additionally, no pH change was observed on both CBCs in the same period. Therefore, it was considered that difference of the amount of organic matter could be influenced to the difference of tendency of the fluoride removal in each sample.

3.5 | Evaluation of fluoride removal capacity of chicken bone chars by shaking test

Figure 7 shows the relationship between fluoride removal capacity, F_{CBC}, and (a) carbon amounts or (b) square of crystallite size. The square of crystallite size was used as a factor of surface area for HAp in the CBC. Figure 7A demonstrated logarithmical (R^2 = .9642) or linear (R^2 = .9341) relation between F_{CBC} and carbon content; however, no simple relations were found on between F_{CBC} and square of crystallite size in Figure 7B. In the previous report,[17] the role of organic substances on CBC was inferred as the inhibitor for fluoride removal because the presence of them on the surface of CBC prevents contact between fluoride with bone minerals. Our results, however, suggested that the presence of carbon on CBC could be positively influenced to F_{CBC}.

On the other hand, Figure 7A illustrates that relation between carbon content and FCBC had larger mismatches in low carbon content region than high carbon content region. The similar trend was also found in low square of crystallite size region in Figure 7B. Further, the CBC0.5 and CBC1 showed very similar in crystallinity (approximately 35 nm as crystallite size for 002 direction), F_{CBC} was drastically decreases from 3.56 mg/g_{CBC} to 2.14 mg/g_{CBC}. Therefore, we hypothesize that (a) generally F_{CBC} is dominant factor to carbon content, and (b) crystallinity also affects F_{CBC} semi-dominantly but does not affect strongly in comparison to carbon content. To confirm them, the region was divided into two parts: One is relatively high carbon content area, the CBC0.5 to CBC6, and the other is relatively low carbon content area, the CBC6 to CBC24. The corresponding regions were also applied to relatively low and high crystallinity (square of crystallite size) samples. Figure 8 shows relations between F_{CBC} and (a) high carbon, for example, (b) low crystallinity region. Linear

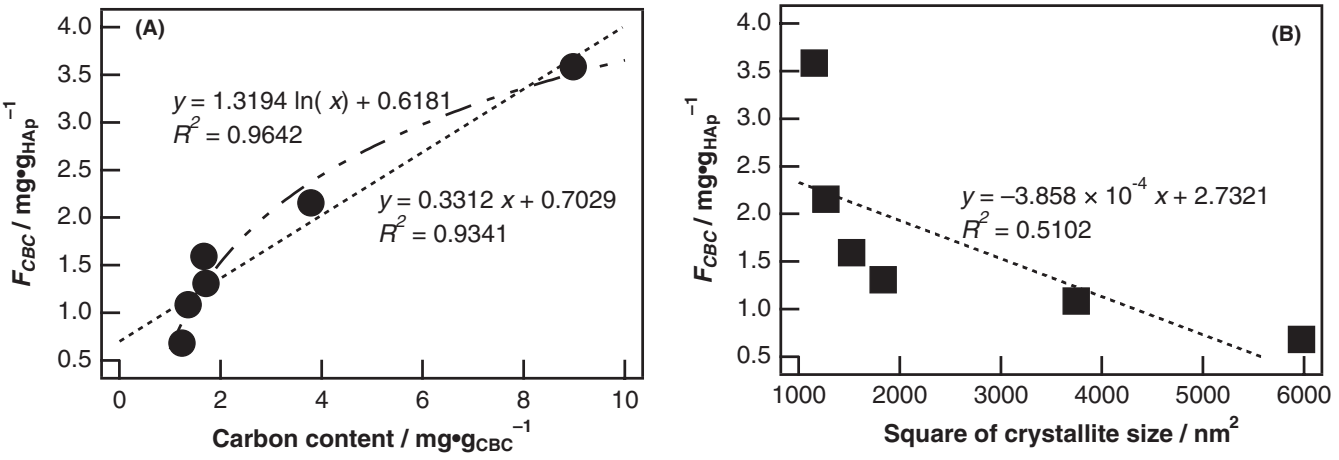

FIGURE 7 Relations between F_{CBC} and (A) carbon content of chicken bone chars (A), and between F_{CBC} and (B) square of crystallite size of chicken bone chars

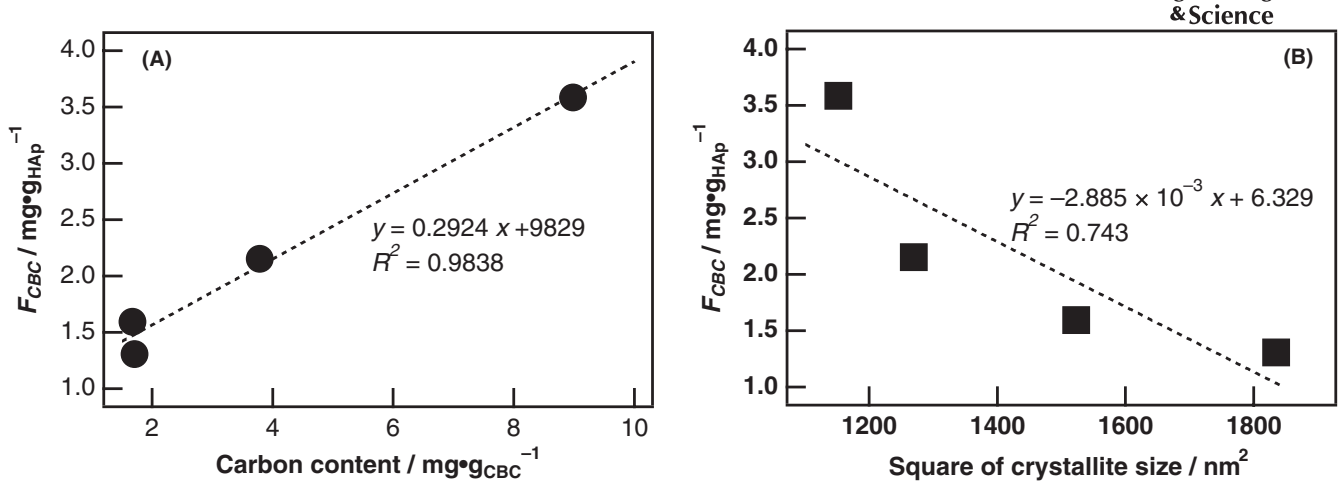

FIGURE 8 Relations between F_{CBC} and (A) carbon content of chicken bone chars in relatively high carbon content region, and between F_{CBC} and (B) square of crystallite size of chicken bone chars correspond to region of (A)

relation with R^2 of .984 was found for F_{CBC}-carbon instead of R^2 of .743 for F_{CBC}-crystallinity. Opposite results were found for relatively low carbon content, for example, relatively high crystallinity region, as a linear relation with R^2 of .816 for F_{CBC}-carbon instead of R^2 of .941 for F_{CBC}-crystallinity as shown in Figure 9A,B, respectively. These results, summarized in Figure 10A,B, suggest that strong relativity exists in between carbon content and F_{CBC} at higher carbon content region but its effect becomes smaller in lower carbon content region. Contrarily, negative tendency between F_{CBC}-crystallinity was found in higher crystallinity region as well as worse tendency for F_{CBC}-carbon. These evaluations are very simple, and much detailed evaluation will be required to elucidate the influence of carbon in the CBC; however, as a first approximation, our hypothesis was confirmed for our present CBC samples. In any case, the present results demonstrated that carbon content in CBC had positive influence on fluoride ion removal, contrary to Larsen et al.[17] Detailed mechanism of carbon effects on fluoride removal has to be investigated; however, the following hypothesis would explain the present results that temporal adsorption and/or capture of fluoride ion by microstructure of carbon in CBC occurred before immobilization it in apatite structure by ion exchange and/or dissolution-precipitation process.

A variety of removal ability of chicken bone char has been studied not only for fluoride removal but also for some toxic ions such as arsenic, lead, and cadmium. Actually, experimental or practical implementation of bone char has planning for the place where facing the natural contamination problem of water. CBC treatment of fluoride ion takes enormous time to satisfy the permissible value of remaining fluoride concentration, and it is required to reduce the reaction time of CBC with fluoride in order to consider the practical use such as for drinking.[13] On the other hand, natural carbonized materials were generally using for the adsorbent by the activation process. Therefore, the activation treatment could be considerable to modify the property

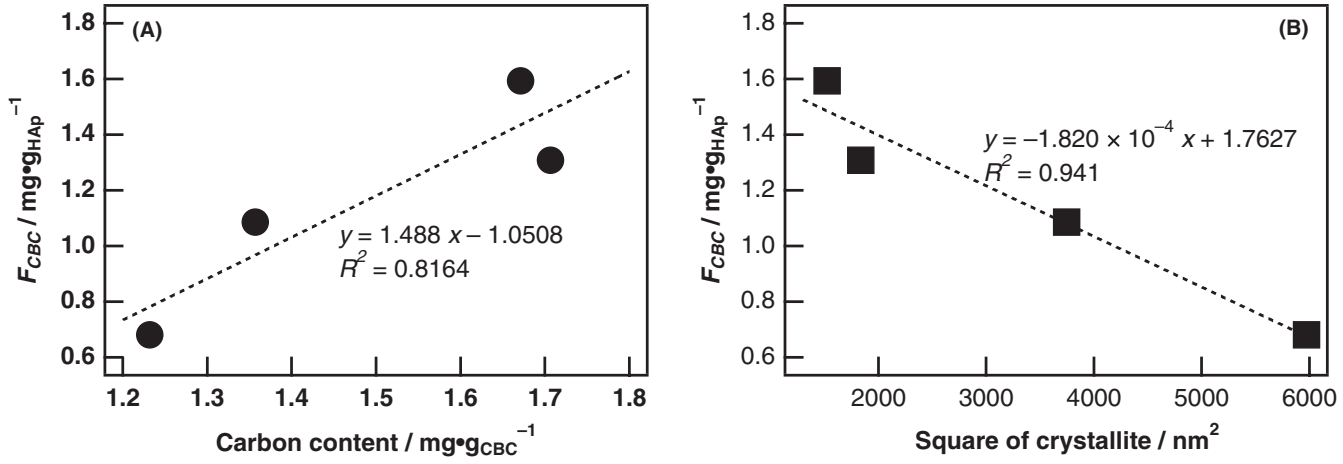

FIGURE 9 Relations between F_{CBC} and (A) carbon content of chicken bone chars in relatively low carbon content region, and between F_{CBC} and (B) square of crystallite size of chicken bone chars correspond to region of (A)

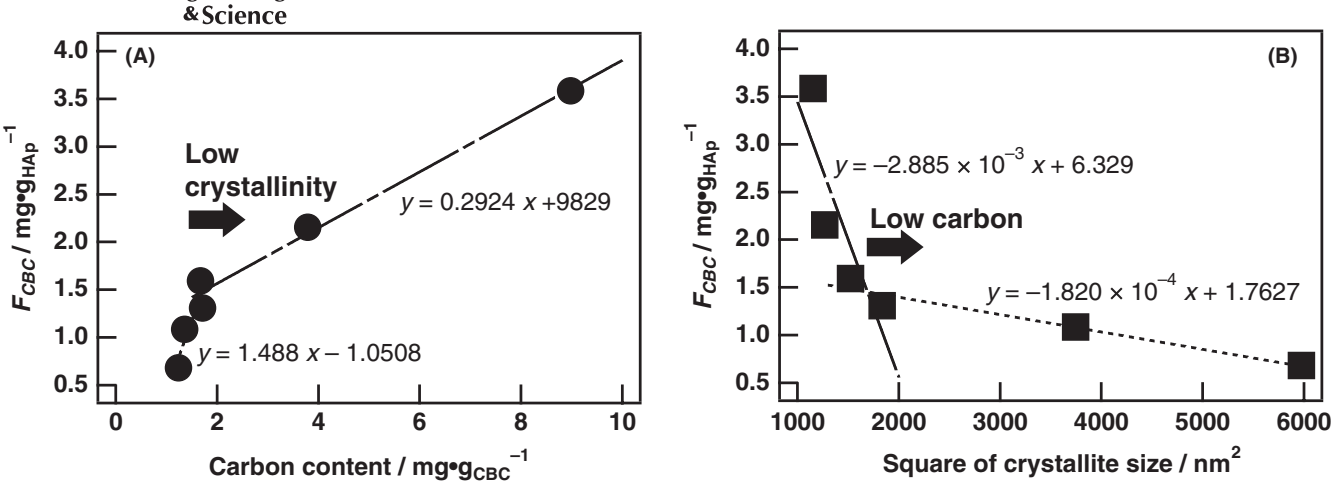

FIGURE 10 Summary of relations between F_{CBC} and (A) carbon content and (B) square of crystallite size (B). These figures demonstrate dominant factors, carbon content, and square of crystallite size in each region

of CBC such as reaction time and removal capacity even in the case of CBC.

4 | CONCLUSION

In this study, to confirm the contribution of the carbon on the CBC for fluoride removal, chicken bone chars with various carbon content were prepared by changing calcination time of chicken bone. Although heating temperature was lower than HAp crystal-grain growth temperature, HAp in chicken bone grew by long heating time. The fluoride removal test demonstrated that carbon content showed higher relativity with F_{CBC} than crystallinity by the HAp crystallite size around 35 nm, approximately 50 crystal unit to *c*-axis, and crystallite size became dominant factor for fluoride removal when the crystallite size was larger than 40 nm. These results suggested that effective carbonization of organic substances concomitant with a maintenance of HAp crystallite size is important to effective removal of fluoride ion.

ACKNOWLEDGMENTS

The authors deeply appreciate the helpful support with the experiments given by Mr. Sakun Preedavijitkul (King Mongkut's Institute of Technology Ladkrabang) and Mr. Yoshiki Kameda (National Institute of Technology, Toyama College). This research was partially supported by KAKENHI, Grant-in-Aid for Scientific Research (A), and Japan Society for the Promotion of Science (JSPS).

ORCID

Masanori Kikuchi https://orcid.org/0000-0002-9451-8147

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How to cite this article: Kikuchi M, Arioka Y, Tafu M, Irie M. Changes in fluoride removal ability of chicken bone char with changes in calcination time. *Int J Ceramic Eng Sci*. 2020;2:83–91. https://doi.org/10.1002/ces2.10034