PREPARATION OF BONE CHAR BY CALCINATION

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SUMMARY: Preparation of bone char in ceramic kilns as used in the northern part of Thailand is normally a calcination rather than a charring process. This laboratory study shows that firing the cleaned bones up to 500°C and then gradually opening the kiln gate and letting it cool down results in bone char of a good quality and a with maximum fluoride removal efficiency of 91.8 %. At higher temperature of calcination, the percentage was less, decreasing with increasing temperature. At 800°C the percentage of fluoride removed was less than 50. For comparison, the efficiency of normal ICOH bone char, which is heated in an electric furnace at 600°C for 20 minutes, was only 69.4 %. Thus calcination in ceramic kiln may be a promising process for use by local communities.

Key words: Bone charring; calcination, local preparation, Thailand.

INTRODUCTION

The ICOH Defluoridator, which has been available for defluoridation in Northern Thailand for several years, is based on bone char burnt at 600°C for 20 minutes in an electric furnace. Its defluoridation properties are attributed to adsorption and ion exchange of the water fluoride. Its efficiency depends on the initial fluoride concentration, the amount of bone char available and amount of water treated and the rate of water flow. The bone media of the ICOH defluoridators have to be changed every 1 - 3 months. Nowadays, ICOH is no longer able to supply ready-made filters to communities and communities cannot produce such a bone char themselves because of the high cost of electric furnaces as well as pollution to the environment of the bone charring process.

In 1995, three high-school children living in high fluoride areas in Lamphun Province tried to solve this problem in their community by charring cattle bones on a stove by the calcination process, but with uncontrolled temperature. They used the black coloured bone char as a filter medium in their defluoridators and measured the residual fluoride in the water after filtration. Their experiences demonstrated good efficiency in fluoride removal.[1] However one disadvantage of this bone char was that the water, after filtration, had a bad smell and was yellow in colour, i.e. unfit for drinking. To treat the bad odour and yellow water, 1 litre of activated carbon was used to make it clear and odourless. This would increase the cost of filtration and necessitate cleaning the activated carbon every 3 - 4 weeks and completely changing it every 3 months. However, the community can carry out this method of bone char production, if the process could be carried out at optimal temperature, providing high removal capacity of the medium and good water quality for drinking. Thus the calcination of bones is an interesting process, which may be the process of choice for communities in their local preparation of defluoridation media.

From many scientific studies reviewed, the two charring techniques are described:

1. **Calcination** is known as a process of high temperature heating in the presence of atmospheric oxygen. The end product being pure bone mineral, a compound related to hydroxyapatite. All organic material are combusted to CO_2.
2. **Pyrolysis** is a charring process with no or very limited access to oxygen. In the organic phase the bone is converted into inorganic carbon and graphite, which makes this bone char black whatever charring temperature is used.

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However, the fluoride uptake is mostly ascribed to the bone mineral phase (apatite).[2,3] Recently, a study presented by Dahi and Bregnhøj [3] showed that when bone has free access to oxygen in the atmosphere (calcination), the defluoridation capacity is drastically reduced when heated at temperatures higher than 550°C. In the same range pH and alkalinity of defluoridated water increases. They found that pyrolysis provides the best bone char defluoridation capacity demonstrated by a linear correlation between surface area and defluoridation capacity.[3] According to Mjengera et al.[4] bone charred at 600°C for a duration of 20 minutes has an optimal fluoride removal capacity.

MATERIALS AND METHODS

Calcination. Twenty kilograms of cattle bone were cleaned, all meat remnants, lipids and tendons being removed, and then dried in the sun for 2-3 days. Batches of 3 kg at a time were heated on the plate of a cross draft kiln with a thermocouple. Atmospheric air was flushed into the kiln chamber from room temperature for approximately 1 hour. Then the firing was stopped and the kiln-gate opened gradually to cool down. The process was performed at 300, 400, 500, 600, 700, 800°C for 1.5 - 2 hours. This yielded 6 batches of bone char weighing 1 kg each. The bone char was ground in a mortar and sieved to obtain 3 - 5 mm grain size.

Conventional calcination. For comparison purposes bone char was also prepared as normally used in the ICOH defluoridator, i. e. calcined up to 500°C, then firing stopped.

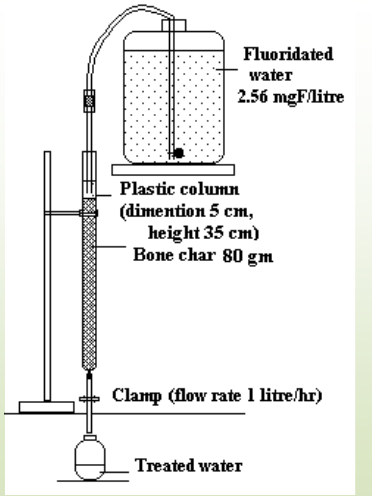

FIGURE 1. Testing column for fluoride removal.

Bone char properties. The colours of the bone char batches were recorded. The defluoridation capability of the bone char batches was tested by placing 80 g in 3 plastic columns imitating defluoridators. The plastic column set-up is shown in Figure 1. The columns were loaded at the rates of 1 or 4 L/h using water containing respectively 2.56 and 2 mg fluoride/L. Treated water samples were collected every 5 respectively 10 minutes of filtration. The fluoride concentrations were measured using Ion Selective Electrode Orion 720A and Orion 720.

Defluoridation capability. The mean residual fluoride for each kind of bone char tested in the columns was calculated. The mean percentage of fluoride removed was used as an expression of defluoridation capability.

RESULTS

Colour of bone char. The colour of bone calcined at the different from low to high temperatures was different, changing respectively from black to brown, grey and white. For each batch the fraction of the different colours was determined, Table 1.

TABLE 1. Physical property of bone calcined at different temperatures

Temp. °C	Bone colour	Property of treated water		
		Colour	Smell	Taste
300	black : brown (4:1)	Dark yellow	Bad odour	Undrinkable
400	black : brown (4:1)	Dark yellow	Bad odour	Undrinkable
500	black : brown (3:1)	Light yellow to clear	None	Normal
600	black : grey (4:1)	Clear	None	Normal
700	black : grey (3:1)	Clear	None	Normal
800	Black : grey : white (3:2:1)	Clear	None	Normal

TABLE 1. Fluoride removal (%) in case of filtration through columns of bone char calcined up to different temperatures.

Bone char 80 g, flow rate 1 L/h, S_0=2.56 mg F/L

Temp (°C)	Fluoride removal %
500	91.8
600	75.0
700	62.5
800	45.3

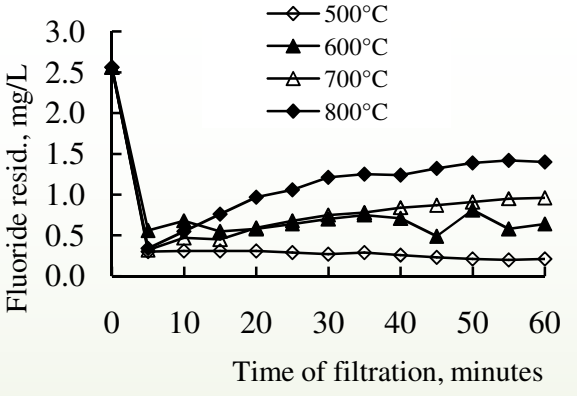

FIGURE 2. Average residual fluoride concentration after filtration by calcined bone char at different temperatures (bone char 80 g, flow rate 1 L/h, S_0=2.56 mg F/L)

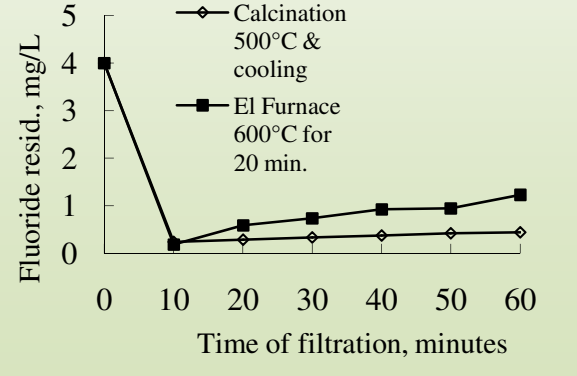

FIGURE 3. Average residual fluoride after filtration by calcined and pyrolysed bone char.

Organoleptic Property of treated water. The appearance as well as the smell and taste of the treated water was evaluated as shown in Table 1.

Defluoridation of water. Bone calcined up to 500°C then stopping firing, had the highest mean percentage of fluoride removed (91.8 %). At higher temperature of calcination, the percentage was less decreasing with increasing temperature. At 800°C the percentage of fluoride removed was less than 50 %, Table 2, Figure 2.

The bone char normally used in the ICOH defluoridators, i.e. heated at 600°C in electric furnace for 20 minutes, was found to remove 69.4 % of the water fluoride.

Thus compared with the "normal" bone char, the bone calcined as described above at 500°C, with 88.9 % removal, had 28 % higher defluoridation capacity.

DISCUSSION

Because the burning bone was also fired by its organic components, it continued burning for a while after the burner had been extinguished in the kiln. During that time the calcination temperature would increase if no heat was released. Hence, to stop the increase of calcination temperature, the kiln gate was immediately and gradually opened after firing stopped.

The results of this study seems to correspond to that of Dahi and Bregnhøj[3] who concluded that the defluoridation capacity is drastically reduced between 550°C and 600°C, if we assume the fluoride removal efficiency shown in this study. However, the optimal calcination temperature presented (500°C) may be different from their experiment, according to different procedures. According to this study however, maximum removal capacity was obtained by firing the bones from room temperature up to 500°C in 1.5 - 2 hours then stopping firing and gradually opening the kiln gate to let the calcined bone char cool down. Moreover, the treated water by calcined bone char at optimal temperature had good properties of the treated water.

Further studies should be carried out to confirm the efficiency and clarify other parameters in defluoridation by calcined bone char, before bringing it to communities as fluoride filter for drinking water. However the results obtained so far seem to show that the calcination procedure followed may be appropriate for production of bone char by local communities

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