



Fabrication of poorly crystalline hydroxyapatite nano-particles by rapid auto-ignition route as efficient adsorbent for removal of disperse blue dye



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ABSTRACT

In the present investigation the poorly crystalline hydroxyapatite was rapidly fabricated by the gel auto-ignition route using calcium nitrate tetra-hydrate and di-ammonium hydrogen orthophosphate as a starting materials and urea as a fuel. The produced powders were employed as adsorbents for uptake of disperse blue 60 from aqueous solution and the adsorption performance was spectrophotometrically studied to determine the appropriate condition for fabrication of amorphous hydroxyapatite. The effects of synthesis parameters such as fuel ratio, gel preparation pH, calcination temperature and the influence of processing factors like dye solution pH and material dosage on dye adsorption have been investigated, comparatively. The properties of products were characterized by X-ray diffraction (XRD), Fourier transformed infrared spectroscopy (FTIR), dynamic light scattering (DLS) and scanning electron microscopy (SEM). The synthesized hydroxyapatite can supply a large quantity of active sites to enhance the uptake of dye. Although, the change in pH of synthesis condition shows a negligible variation in the adsorption efficiency, the acidic condition leads to formation of β -tricalcium phosphate. The stronger adsorption as well as the higher activity with disperse blue 60 was observed in pH of 2.0. In addition, the adsorption was found to be controlled by the calcination temperature. Furthermore, it was indicated that the dye adsorption on poorly crystalline hydroxyapatite prepared in the appropriate condition, fuel ratio of 0.50 and pH of 7.0, is well adjusted with Langmuir isotherm in which the maximum adsorption capacity was determined to be 4600 mg g^{-1} , approximately.

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1. Introduction

Hydroxyapatite, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, is one of the most important member of calcium phosphates which found several applications in the bio-ceramics because of excellent biocompatibility and compositional similarity to the human bone [1–3]. Over the recent decade, hydroxyapatite, HA, is found the other variety of industrial purposes in chromatography [4,5], heterogeneous catalyst [6,7] and adsorption for uptake of heavy metal ions [8–10]. HA is most desirable material for employment as adsorbent due to high surface area and porous structure [3,11]. The characteristics of HA as an adsorbent are largely governed by the crystallinity, particle size, morphology, homogeneity and porous structure which can be

controlled by fabrication techniques [12–14]. However, the application of conventional fabrication techniques such as solid-state reaction is rather difficult for manufacture of homogenous HA [11]. On the other hands, the solid state reactions generally cause the formation of other crystalline phases such as β tri-calcium phosphate, β -TCP, and calcium oxide which cannot be transformed to a stable HA by heat treatment [15,16].

Up to date, various wet chemical methods have been developed and surveyed to fabricate nano-sized amorphous HA, including sol-gel [17,18], hydrothermal [19,20], co-precipitation [21–23] and emulsion [24,25]. In order to develop the application of HA in wastewater treatment, it is necessary to explore an available route to achieve the HA particles in nano-scale. Auto-ignition is perhaps one of the most popular methods applied to rapid manufacture of poorly crystalline materials [26]. The major advantage of this method is high throughput for producing a product with high purity [11,27]. Auto-ignition is a very simple technique in which an

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aqueous solution containing the desired metal nitrates and fuels are simultaneously heated to form a complex gel and then the obtained mixture undergoes self-ignition by initial heat treatment. The exothermic propagating reaction leads to formation of a fluffy material [28]. The favor amorphous phase with high purity can be produced in only one step and no further calcination maybe required in comparison to other wet methods. Commonly nitrates are greatly used as oxidizer and urea [29], glycine [28,30] and citric acid [31] have been used as fuels in the auto-ignition method. Urea is well known as appropriate organic fuel for combustion because, it sufficiently generates local heat to complete combustion reaction and creates gaseous phase to inhibit particle agglomeration, simultaneously [26,32]. Auto-ignition has several advantages in fabrication of HA, for instance, providing high specific surface area and good homogeneously distributed particles, control of crystallinity, avoiding produce other undesirable calcium phosphate phases [33,34].

With the growth of industrialization, the organic disperse dyes become one of the most significant pollutant sources which discharged through the environment by textile, tannery and food industries [35–37]. The presence of dyes even in a very low content especially, in water resources causes adverse effect on human and marine creature health [38]. In addition to change the water color by organic dyes, the dissolved oxygen is declined and sunlight is prohibited to diffuse into the deep regions and photosynthesis is depleted, consequently. Therefore, it is great of importance to remove dyes from wastewater by appropriate separation and purification techniques [39,40]. Several physical, chemical and biological techniques like coagulation [41,42], flocculation [43], precipitation [44], membrane filtration [45,46], electrocoagulation [47] and biological degradation [48] are developed to remove contaminants which frequently are not applicable for removal of dyes from wastewater. Adsorption is one of the preferred techniques for recovery and recycling of dyes from wastewaters due to low cost, ecofriendly, simplicity and high efficiency [49,50]. Various conventional and unconventional adsorbents such as clays [51,52] activated carbon [53,54], zeolite [55] and biomaterials [56,57] have been applied for uptake of dyes. Industrial dyes have different chemical nature and can be classified as disperse, reactive, acid, basic, direct, azoic, sulfur, and direct dyes [37]. Reactive, direct, basic and acid dyes are soluble in water and can be removed from effluent by adsorption on natural clays like ion-exchange adsorbents [58] and magnesium oxide [59]. Although, HA cannot effectively remove these dyes, the effluents contaminated by disperse dyes can be efficiently treated by HA [39]. As a result, it is important to discover a novel adsorbent with easy preparation, high yield, low cost and great adsorption efficiency for removal of dyes from wastewater. Although, HA have been widely used as adsorbent for wastewater purification contaminated by heavy metal ions [60–64] due to nontoxicity and biocompatibility but this biomaterial has been rarely investigated for removal of dyes [65–67].

There are a few articles for dyes removal by HA in the batch and dynamic system [68]. Barka et al. [39] used the HA synthesized by precipitation method for disperse blue SBL removal. Lemlikchi et al. [69] reported that the adsorption of textile dyes on HA is carried out, heterogeneously. Allam et al. demonstrated that the HA prepared by a combined precipitation-microwave method is efficient for removal of methylene blue from aqueous solution and the microwave irradiation enhances the uptake of dye [70]. Recently, Lemlikchi et al. synthesized HA in blue hydron solution by precipitation method to treat the contaminated water [71].

Auto-ignition can be an alternative fast technique for fabrication of poorly crystalline HA powder with the controlled stoichiometric composition. To our knowledge, a few investigations were performed on the auto-ignition synthesis of HA and their applications

in adsorption of disperse dyes. In the present work, HA powder was synthesized by the auto-ignition route and the as-produced powder was directly used in the treatment of aqueous solution contaminated by disperse blue dye 60. The physico-chemical properties of powders were characterized to well understand the role of fabrication factors such as fuel ratio, calcination temperature and synthesis pH on dye removal. Moreover, the adsorption performance of obtained HA powder was comparatively investigated in aqueous solution with different pHs. Finally, the influence of powder characteristics on wastewater treatment is discussed in details.

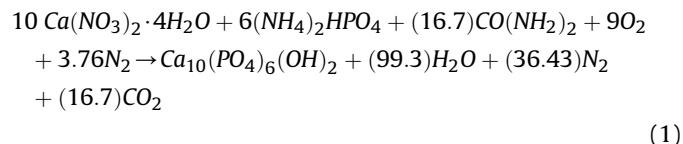
2. Materials and experimental methods

2.1. Materials

Calcium nitrate tetra-hydrate, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, as a calcium source, di-ammonium orthophosphate, $(\text{NH}_4)_2\text{HPO}_4$, as a phosphorus source and urea, $\text{CH}_4\text{N}_2\text{O}$, as a fuel were obtained from Merck Company. All reagents were analytical grade without further purification. Distilled water was used in experiments and the pH of solutions was controlled by hydrochloric acid, HCl, 37.0%, ammonium hydroxide, $\text{NH}_3 \cdot \text{H}_2\text{O}$, 25%. Moreover, disperse blue 60, $\text{C}_{20}\text{H}_{17}\text{N}_3\text{O}_5$, (India Dye Chem) was employed as a colorant agent.

2.2. Preparation of powders

In order to synthesize 2.00 g of stoichiometric HA powder according to the following reaction, aqueous stock solutions were prepared by dissolution of 4.72 calcium nitrate and 1.57 g di-ammonium orthophosphate, DAP, in 10 ml distilled water, separately.



Different contents of urea, in which the fuel ratio was considered to be 0.125, 0.250, 0.500, 1.00 and 1.500 times of stoichiometric value, were first dissolved in 10 ml water with continuous stirring and then added to calcium nitrate solution. In the second step, DAP was added dropwise to the calcium nitrate-urea solution with quick stirring and pH was continuously controlled by adding appropriate content of hydrochloric acid and ammonium hydroxide at different levels, from 1.0 to 9.0. The solutions were heated at 100 °C to slowly form a gel with adjusting pH at desired level at the same time. The obtained gels were transferred into preheated furnace, 500 °C, where the ignition took place and fragile foams were produced at 7 min. In order to evaluate the effect of crystallinity on adsorption, the fragile foam was easily powdered and calcined at 500, 600, 700 and 800 °C.

2.3. Batch adsorption experiments

In order to evaluate the dye removal ability of prepared powders, batch adsorption experiments were performed and the effects of calcination temperature, synthesis pH, adsorbent dose, dye solution pH and fuel ratio were examined on the adsorbent performance. The colorant solutions were prepared by mixing different contents of disperse blue dye with distilled water in which the dye concentrations were 2000, 2500, 3000, 3500, 4000, 4500 and 5000 mg l^{-1} . The synthesized powders, 5–10 mg, were dispersed in 10 ml water and the pH was adjusted from 1 to 5 using HCl solution,

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