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PERFORMANCE EVALUATION OF HYDROXYAPATITE PREPARED FROM EGGSHELLS IN CARBON DIOXIDE ADSORPTION

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Abstract: Eggshell waste is typically produced from daily poultry consumption and industrial applications. They are a rich source of calcium in the form of carbonates and oxides, recognised as excellent hydroxyapatite sources (HAp). To date, limited studies have highlighted the modification of HAp with impregnation. In the present study, HAp was prepared via the precipitation method, and further modification of HAp using monoethanolamine (MEA) and deep eutectic solvent, particularly choline chloride:urea (ChCl:U), were explored for carbon dioxide (CO_2) capture. The morphological structures were studied using a scanning electron microscope, while properties were assessed using energy-dispersive X-ray spectroscopy techniques (SEM-EDX) and Brunauer-Emmett-Teller (BET). The CO_2 adsorption performance using raw and impregnated HAp was also evaluated. By introducing the chemisorption process, the impregnated ChCl:U-HAp with irregular crystallite agglomerates demonstrated a higher adsorption capacity and longer breakthrough time than raw HAp and MEA-HAp. This study confirms the feasibility of using eggshells to produce HAp as an effective adsorbent in CO_2 capture.

Keywords: Hydroxyapatite, eggshell, carbon dioxide adsorption, impregnation, deep eutectic solvent

1. Introduction

In the era of industrialisation, the combustion of fossil fuels is the major culprit of environmental issues like climate change and global warming. In compliance with the Kyoto Protocol and the Paris Agreement, the scientific community has proposed several techniques in carbon capture, utilisation and storage (CCUS) to combat global warming. Some CCUS techniques, such as chemical absorption, membrane separation and cryogenic processes, have been widely implemented to minimise carbon dioxide (CO_2) emissions (Zhao et al., 2018). Chemical absorption using amines is commonly used in industries due to high CO_2 capture efficiency. However, it encounters high energy consumption during regeneration and experiences solvent degradation problems (Azmi & Aziz, 2019). Therefore, adsorption using porous carbonaceous materials is more attractive, with the advantages of low-cost synthesis and high thermal and chemical stability. To achieve satisfaction in aspects of operational and economical, novel adsorbents have been produced from low-cost natural wastes such as tea (Rattanaphan et al., 2020), municipal solid waste (Karimi et al., 2020), walnut shell (Khoshraftar & Ghaemi, 2022) and other biomass sources (P. H. Ho et al., 2021).

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The yearly generation of about 250,000 tons of eggshells from daily use results in landfill disposal without further processing, leading to other environmental issues, such as disgusting smell and infection propagation risk (Mignardi et al., 2020). Hence, reusing and converting eggshell wastes into useful materials for sustainable development is vital. In recent years, eggshell wastes have been utilised as a low-cost adsorbent due to their porous structure. Several researchers highlighted that eggshells have a high specific surface area that aids in heavy metal removal (Abatan et al., 2020; Latiff et al., 2022; Tizo et al., 2018). Considering the calcium levels in eggshells, they could serve as a potential source for synthesising hydroxyapatite (HAp) . HAp is an organic mineral with a typical $Ca_{10}(PO_4)_6(OH)_2$ lattice structure. Due to its calcium and phosphate content (the main mineral component of bones and teeth), it offers several applications, including bone repair and tissue regeneration (Tangboriboon et al., 2019). HAp is also gaining significant attention in developing novel adsorbents due to their favourable properties, such as high adsorptive, thermal and chemical stability (J.-Y. Ho et al., 2024).

A deep eutectic solvent (DES) is a eutectic mixture with two or three components capable of self-dissociating to form a eutectic solvent of a lower melting point than their individual (Chia et al., 2023). DES is formed by a hydrogen-bond donor and a hydrogen-bond acceptor components via hydrogen bonds. DES is widely recognised as a green solvent as it offers several advantages, such as low toxicity, low volatility, low flammability, high solubility capacity, high thermal stability, and biodegradable properties (Babaei & Haghtalab, 2023; Massayev & Lee, 2022). Recently, many efforts have been made to develop DES for CO_2 capture. Trivedi et al. (2016) synthesised monoethanolamine hydrochloride-ethylenediamine DES for CO_2 capture. The DES

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could interact with CO_2 gas to form carbamate. An adsorption capacity of 0.54 mol CO_2/mol solvent was reported for the system.

Nevertheless, little scientific research on CO_2 adsorption using DES-impregnated HAp is available. Therefore, this work would explore eggshell-based HAp to be impregnated with DES (particularly choline chloride:urea, ChCl:U) as a bioadsorbent for CO_2 capture. A comparison study was also included by impregnating the HAp with monoethanolamine (MEA), a common solvent used for CO_2 capture. The results of CO_2 adsorption performance were supported by scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) and Brunauer-Emmett-Teller (BET) analyses.

2. Methodology

2.1 Materials

All chemicals and reagents were in analytical grade and used without further purification. Phosphoric acid (H_3PO_4, 85%) and urea (CH_4N_2O, 99%) were purchased from Anhui Fulltime. Nitric acid (HNO_3, 69%) and ammonium hydroxide (NH_4OH, 25%) were supplied by R&M Chemicals. Ethanolamine (C_2H_7NO, 99%) and choline chloride ($C_5H_{14}ClNO$, 99%) were purchased from Thermo Fisher Scientific; meanwhile, ethanol (C_2H_5OH, 95%) was from HMBG.

2.2 Pretreatment of Eggshells

All the collected eggshells were cleaned with water to remove impurities, followed by 30 min of boiling and removing their inner white membrane. The eggshells were sent into an oven (AX30, Carbolite, UK) for drying overnight. Dried eggshells were ground into fine powder using an agate mortar and passed through a 10 mesh sieve, followed by calcination in a furnace (DAIHAN FHX-14, Daihan Scientific, Korea) at a temperature of 900℃ for 4 h.

2.3 Synthesis of Hydroxyapatite (HAp)

Pretreated eggshells were reacted with HNO_3 (69%) at a weight ratio 10:6 under constant stirring. Titration was done by adding 50 ml of 0.6 M H_3PO_4 solution into the mixture. The pH of the mixture was maintained at 10 using NH_4OH solution. The mixture was put aside for precipitation, and the resultant precipitate was filtered and washed using distilled water and ethanol. The washed sample was sent for overnight drying, followed by calcination at 900℃ for 1 h to form HAp powder.

2.3 HAp impregnation with MEA and DES

HAp was impregnated with MEA according to the procedure introduced by Khalil (2018). 2 g of MEA was stirred with 5 g of HAp for 1 h, and deionised water was added as an environmentally friendly medium to facilitate the impregnation process. The slurry was filtered and dried in the oven at 70°C for 6 h to form MEA-HAp. Choline chloride and urea were mixed in a molar ratio of 1:2 at 80℃ with constant mixing until homogenous. The homogenous solution developed is known as DES. Impregnation of HAp with DES was fixed at a weight ratio of 1. 5 ml ethanol was added to HAp and DES, followed by 2 h stirring to ensure complete impregnation. The mixture was kept overnight and dried at 80℃ for 6 h, forming DES-HAp.

2.4 Characterisation Studies and Adsorption Test

The HAp samples were sent for scanning electron microscopy (SEM) characterisation (Vega3, TESCAN, Czech Republic) to study the surface morphology of the synthesised bioadsorbents. Energy dispersive X-ray spectroscopy (EDX) (EDX-Oxford, TESCAN, UK) were incorporated to analyse the elemental composition of the adsorbents. The Bunauer-Emmett-Teller (BET) method was used to study samples' specific surface area using Micromeritics Tristar II Plus, USA.

The CO_2 adsorption study was conducted using a packed bed column to determine the adsorbent's performance in CO_2 capture. Breakthrough curve and maximum CO_2 adsorption capacity were determined. Initially, 30 g of the bioadsorbent was transferred into the packed bed column. The column was purged with N_2 gas for 10 min at room temperature and atmospheric pressure to create an inert environment. The adsorption study was conducted by introducing a mixture of N_2 and CO_2 gases into the packed bed column, in which the CO_2 composition was maintained at 15% under a constant flow rate of 200 ml/min using a digital mass flow controller. A CO_2 analyser (Alpha Omega series 9610) was connected to the column outlet to analyse the CO_2 concentration. The results were collected and recorded using a data logger system (GRAPHTEC GL820).

3. Results and discussion

3.1 SEM Characterisation

SEM was used to identify the surface morphology of HAp samples. Figure 1 shows the SEM images of raw HAp, MEA-HAp and DES-HAp under the magnification of 3000×. Raw HAp shows smooth flakes of porous surface (Figure 1(a)). The observed morphology could be effective in CO_2 adsorption. Unigul and Nigiz (2020a) reported that activated carbon exhibited a smooth and porous structure that demonstrated a higher CO_2 adsorption than other samples. Therefore, the similarity of HAp morphology to that of activated carbon could suggest the effectiveness of the HAp in CO_2 adsorption. Impregnation was introduced to modify the surface properties of the HAp for enhanced CO_2 adsorption capacity. With impregnation, an irregular cloud-like shape and more visible pores were observed in MEA-HAp (Figure 1(b)), and irregular clouds with distinct pores were observed in DES-HAp (Figure 1(c)). The distinct pores could provide more active sites for CO_2 adsorption, as Makoś and his co-authors (2020) suggested.

3.2 EDX Characterisation

EDX analysis was performed to identify the elemental composition of the HAp samples. Table 1 summarises the elemental composition of carbon (C), oxygen (O), calcium (Ca), phosphorus (P), nitrogen (N) and chlorine (Cl) in each sample. Figure 2(a) reveals that the typical elements of C, O, Ca and P were found in the raw HAp. The presence of C evinced the synchronised process. Heat supply during the calcination process initiated the

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Malaysian Journal of Science 43 (Special Issue 1): 44-49 (July 2024)

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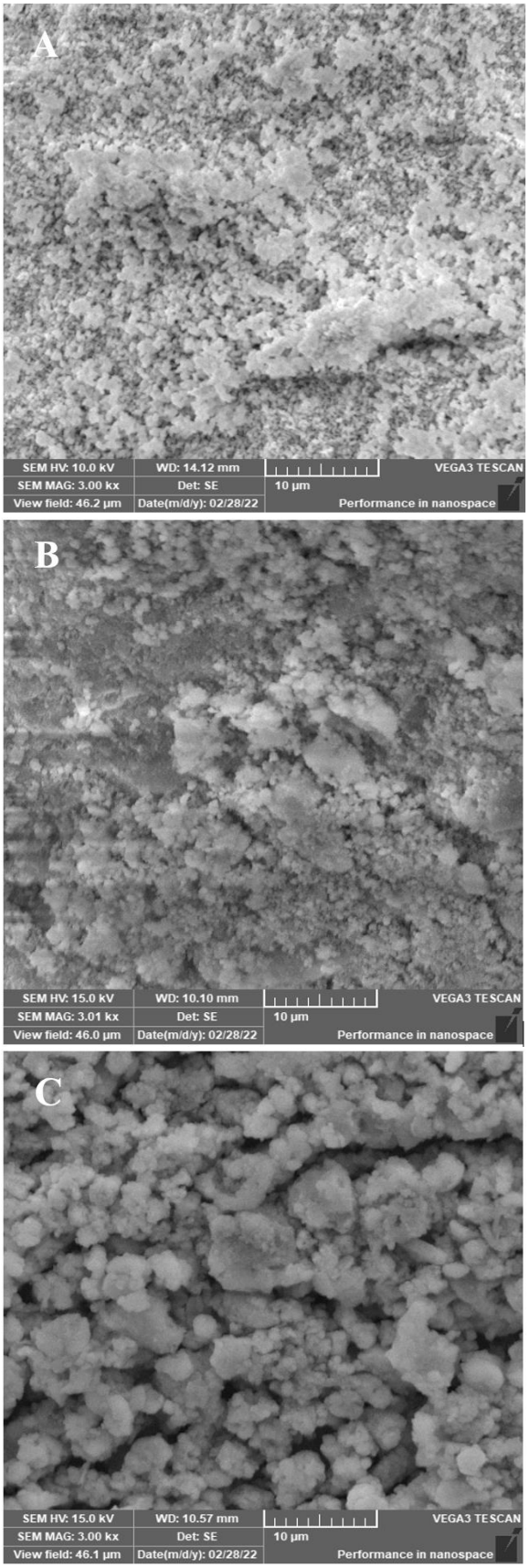

Figure 1. SEM image of (A) raw HAp, (B) MEA-HAp and (C) DES-HAp.

Table 1. Elemental composition of raw HAp, MEA-HAp, DES-HAp

Element	Weight (%)		
	Raw HAp	MEA-HAp	DES-HAp
C	7.04	10.35	21.19
O	58.34	44.43	36.13
Ca	21.09	24.75	20.87
P	13.53	12.74	11.08
N	-	5.77	9.49
Cl	-	1.96	1.23

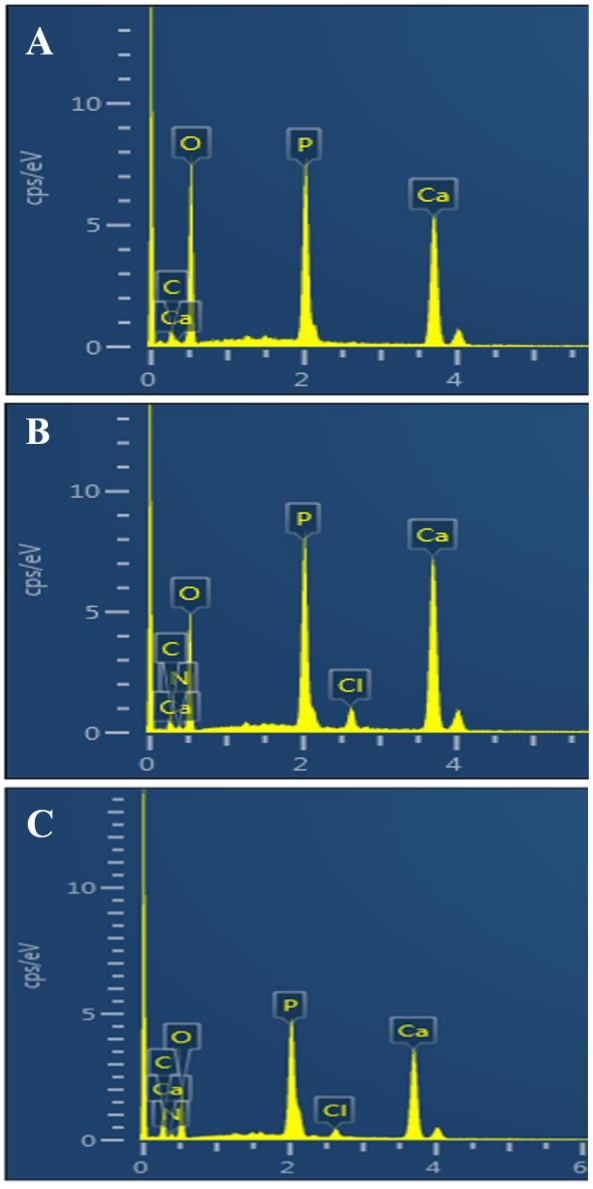

Figure 2. EDX spectra of (A) raw HAp, (B) MEA-HAp and (C) DES-HAp.

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Malaysian Journal of Science 43 (Special Issue 1): 44-49 (July 2024)

volatile matter from the eggshells. Subsequently, it produced a stable carbon element in the raw HAp (Agbabiaka et al., 2020). The element P was detected due to the addition of phosphoric acid during the titration process in the HAp synthesis reaction (Ariyanto et al., 2021). Additional elements of N and Cl were detected in MEA-HAp and DES-HAp samples (as shown in Figures 2(b) and 2(c))due to the impregnation solvents of MEA and ChCl:U, respectively. The presence of N provided more active sites for CO_2 adsorption. The features stem from the inherent qualities of the N element, allowing it to bind effectively with CO_2 based on its acidity.

3.3 BET Characterisation

Table 2 presents the BET surface area and pore characteristics of the raw HAP, MEA-HAp and DES-HAp. Raw HAp possessed a low specific surface area of 5.71 m^2/g. The result is aligned with the morphology structure discussed earlier in Section 3.1. As shown in Table 2, the specific surface area of MEA-HAp increased significantly from 5.71 m^2/g to 105.84 m^2/g. This sharp change shows that MEA impregnation enhanced the formation of a porous structure, directly increasing the BET surface area. This observation was also synchronised with the morphology study in Section 3.1, whereby more visible pores were observed in MEA-HAp compared to raw HAp. Nevertheless, the total surface area and pore volume of HAp impregnated with ChCl:U were lower than raw HAp. The total surface area and pore volume reduction in the DES-HAp sample were caused by pores blockage of HAp with DES solvent during the impregnation process (Ghazali et al., 2020).

Table 2. BET surface area and pore characteristics of raw HAp, MEA-HAp and DES-HAp

	RAW HAp	MEA-HAp	DES-HAp
BET(m^2/g)	5.7058	105.8397	5.4287
Langmuir(m^2/g)	26.1995	423.8743	18.6011
Average pore diameter (nm)	0.92873	0.81247	0.71154

3.4 CO_2 Adsorption

The CO_2 adsorption performances were recorded at 273 K and 1 atm, and the results were presented in Figure 3 and Table 3. All samples followed a similar adsorption trend. The CO_2 uptake of all samples increased with contact time and occupied all available adsorption sites until a final equilibrium was obtained. However, each adsorbent had a different time spent to reach saturation. Raw HAp demonstrated an adsorption capacity of 5.14 mg/g at 3.05 mins breakthrough time. As presented in the SEM morphology study in Section 3.1, raw HAp possessed a large pore diameter, which is advantageous for CO_2 to diffuse and adsorb into the adsorbent. However, the low adsorption capacity of raw HAp was mainly attributed to its lowest specific surface area, as discussed in BET analysis, which limited the amount of CO_2 adsorbed on the adsorbent.

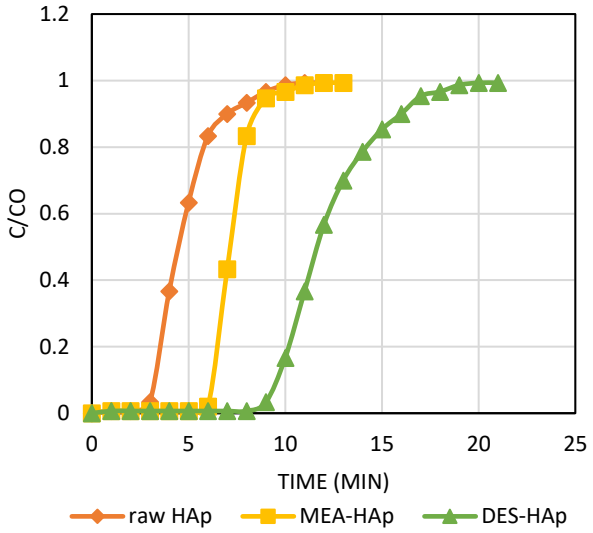

Figure 3. Adsorption breakthrough curves of raw HAp, MEA-HAp and DES-HAp.

Table 3. Breakthrough time and adsorption capacity of raw HAp, MEA-HAP, DES-HAp

	RAW HAp	MEA-HAp	DES-HAp
Adsorption capacity (mg/g)	5.14	10.24	15.38
Breakthrough time (min)	3.05	6.07	9.13

Impregnated HAp demonstrated higher adsorption capacities and longer breakthrough times than raw HAp. MEA-HAp achieved 10.24 mg/g adsorption capacity at a breakthrough time of 6.07 mins. Its excellent absorption quality was highly related to the change of its surface morphology during the impregnation process. High surface area and good porosity after impregnation improved the adsorption performances of HAp, which was also aligned with the finding reported by (Ghosh et al., 2019). Furthermore, impregnation with MEA introduced the amine group, which promoted basic adsorption sites and CO_2 adsorption (Das & Meikap, 2018).

DES-HAp demonstrated the highest adsorption capacity and breakthrough time among all the samples investigated. It manifested 15.38 mg/g with a breakthrough time of 9.13 min. This observation was repudiated with the low surface area and pore diameter finding, as reported in Section 3.3. The improved CO_2 adsorption could be elucidated by introducing active basic sites with ChCl:U impregnation, promoting chemisorption when reacted with Lewis acidic CO_2 (Raja Shahrom et al., 2019). The findings aligned with the work conducted by Zohdi and his co-authors (2019), where chemisorption was observed through the formation of ammonium carbamate. Unlike raw HAp, which depends highly on physisorption and is mainly controlled by the porosity and surface area of the adsorbent, DES-HAp introduced

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Malaysian Journal of Science 43 (Special Issue 1): 44-49 (July 2024)

chemisorption through the introduction of basic sites of N. In chemisorption, CO_2 was adsorbed on the adsorbent via chemical bonds and, therefore, independent of the pore characteristics of the adsorbent.

The CO_2 adsorption capacity of DES-HAp obtained in this study is comparable to other reported literature. For instance, Das and Meikap (2018) reported that activated carbon impregnated with MEA had an adsorption capacity of 12.8 to 18.2 for CO_2 gas. In another work by Ariyanto and his co-authors (2021), the CO_2 adsorption capacity of DES-impregnated activated carbon was found in the range of 13.3 to 15.4. These suggest that the DES-HAp produced in this work offers attractive and competitive performance in terms of CO_2 uptake with other impregnated porous adsorbents.

4. Conclusion

In this study, raw HAp was successfully synthesised from eggshells via precipitation. Impregnation modified the porosity of Hap and introduced amine elements. Impregnation using MEA increased the BET surface area due to enhanced porosity. Meanwhile, impregnation using DES caused a reduction in the BET surface area. High porosity improved the adsorption capacity of MEA-HAp when compared with raw HAp. Despite the lower BET surface area, DES-HAp offered the highest adsorption capacity with the longest breakthrough time. These favourable properties are attributed to the presence of amine group, which promotes chemisorption. The findings suggested that ChCl:U-impregnated HAp could be a potential adsorbent for CO_2 capture. The reusability of the adsorbent can be evaluated in future work to increase the economic feasibility of the process.

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