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# From microporous to mesoporous mineral frameworks: An alliance between zeolite and chitosan

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# Highlights

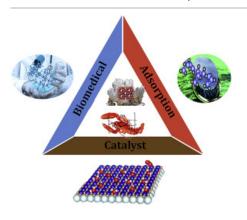
- · Chitosan-Zeolite composites exhibit synergic properties.
- Chitosan-Zeolite composites have been used in various application from separation to biomedical.
- Underlying mechanisms of chitosan-zeolite performance in various application are presented.
- Challenges, limitations, and future directions of the chitosan-zeolite applications are outlined.

#### **Abstract**

Microporous and mesoporous minerals are key elements of advanced technological cycles nowadays. Nature-driven microporous materials are known for biocompatibility and renewability. Zeolite is known as an eminent microporous hydrated aluminosilicate mineral containing alkali metals. It is commercially available as adsorbent and catalyst. However, the large quantity of water uptake occupies active sites of zeolite making it less efficient. The widely-used chitosan polysaccharide has also been used in miscellaneous applications, particularly in medicine. However, inferior mechanical properties hampered its usage. Chitosan-modified zeolite composites exhibit superior properties compared to parent materials for innumerable requests. The alliance between a microporous and a biocompatible material with the accompaniment of negative and positive charges, micro/nanopores and proper mechanical properties proposes promising platforms for different uses. In this review, chitosan-modified zeolite composites and their applications have been overviewed.

# Graphical abstract





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Keywords

Zeolite; Chitosan; Adsorption; Catalyst; Biomedical; Microporous

#### 1. Introduction

Back in 1756, for the first time, natural zeolite was discovered as a potentially large resource. Since then, natural zeolite has been quickly developed and mined from discovery and extraction stages to the vast production in most parts of the world [1]. However, nowadays, it can be synthesized in various ways to customize its characteristics for particular uses. In the basic gel and hydrothermal/solvothermal conditions, zeolites are mostly fabricated from metal cations or organic amines/ammonium cations as the templates or structure-directing agents [2]. Although classical strategies for manufacturing of hierarchical zeolite using mesoporous templates through post-modification are often described as high energy consumption way associated with uses of expensive eco-destructive organic solvents, these days, new protocols of green synthesis have been also designed to effectively synthesize hierarchical zeolites with reducing waste and cost [3].

Natural/synthetic zeolites, as the microporous materials, have been vastly used in many fields including environmental engineering and biomedical engineering applications. To be more specific, the zeolite family was considered in gas separation, organic solvent purification, water, and wastewater cleanup at very low partial pressures and appeared useful desiccants [4]. Purifying air streams for purging volatile organic compounds by the use of zeolite has also been reported [5]. The porous structure has given zeolite new dimensions to be applied in selective adsorption of materials such as organic molecules and heavy metals, gas separation and gas sensing, allowing it to sieve molecules with specific dimensions [6,7]. For instance, zeolite 4A based on coal fly ash was utilized in the removal of heavy metals ions from Refs. [8,9]. It was reported that Ni<sup>2+</sup> and Cu<sup>2+</sup> were removed from wastewater using cellulose acetate/zeolite fibers [10]. However, the high water uptake characteristic of zeolite necessitates the use of a complement to achieve novel platforms with mesoporous structures by the use of biopolymers [[11], [12], [13]], epoxy [14,15], and metal [[16], [17], [18]].

Incorporation of biopolymers into zeolite or in situ synthesis of zeolite in the presence of biopolymers made possible development of mesoporous zeolite frameworks for tailor-making mineral catalysts for biomedical applications. Typically, chitosan [19,20], agarose [[21], [22], [23]], starch [[24], [25], [26]], gelatin [27], alginate [28], silk [29], poloxamer [30], and polylactic acid (PLA) [31]biopolymers have attracted significant attention over the years because of their eco-friendly and biocompatibility features. Chitosan, as a well-known biopolymer, has been utilized in different applications because of its functional groups and biocompatibility [32,33]. However, chitosan has some drawbacks such as colloid formation in water, low surface area, dissolution in acidic solutions and degradation. Cross-linking of chitosan enhances its mechanical properties for acidic media. Cross-linked

chitosan-modified zeolite frameworks are mesoporous networks with superior surface area and efficiency with respect to zeolite alone.

Recent years have seen an increasing number of studies focusing on the uses of bio-based zeolite composites. There have been some reliable reports on the synergism between chitosan and zeolite for engineering applications [34]. Despite such promising features, reports on chitosan-assisted zeolite are scattered and required to be classified. This paper is an overview of chitosan/zeolite frameworks, their synthesis and applications to pave a classical way for designing novel composites with extraordinary characteristics.

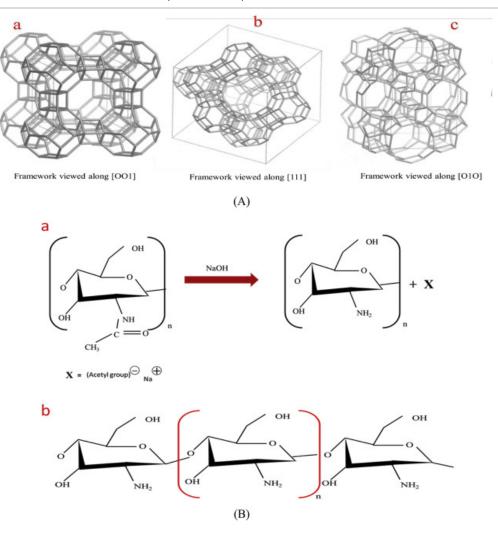
#### 2. Zeolite, chitosan, and zeolite-chitosan materials

In general, zeolites are known as crystalline aluminosilicate porous minerals in which oxygen atoms are arranged in tetrahedral atom structures providing it an extraordinary character for adsorbent, catalyst, and membrane usages. The oxide structure forms an open framework known as tectoaluminosilicates with the possibility of having more than 240 synthetic and 40 natural zeolites that have been identified [35,36]. The Structure Commission of the International Zeolite Association (IZA) has introduced the biggest database of zeolite materials with approximately 250 types which have been coded with a three-letter (e.g., IRR, UWY, JST, etc.).

Notwithstanding their natural origin, zeolites can be synthesized via different methods, including hydrothermal [37], sol-gel [38], and microwave [39]. Hydrothermal and sol-gel are the most prevailing approaches for zeolite synthesis. It is a proven fact that microwave synthesis methods are considered as the most facile approach for zeolite synthesis. Hydrothermal fabrication method, as a subclass of conventional heating methods, is based on transferring the heat from the surface to the middle of the materials. However, the microwave approach could directly transfer heat more intensively, homogenously, and quickly [40]. Zeolites have already utilized in various application such as petrochemicals synthesis; water and wastewater treatments; air separation; detergents; antibacterial agents; etc. due to their cost-efficient, high specific surface area, structural characteristics like a negatively charged lattice, ion-exchange capacity, selectivity, non-toxic properties, ease of availability, and abundantly. Zeolites have been originally utilized as an ion-exchange bed in water purification/softening, separation substrate, catalyst, and sorbent [[41], [42], [43]]. The first generation of synthetic zeolite contained small pores, later a variety of and large-cage zeolites were engineered [44,45]. Attention was also paid to control the pore size and functionality of zeolite frameworks for its higher adsorption capacity [46,47]. Inspired by natural zeolites, zeolitic imidazolate frameworks (ZIFs), a subgroup of pretty new material called metal-organic frameworks (MOFs), including tetrahedral metal cations as the role of silicon and the imidazolate as linkers replaced the role of oxygen have been introduced. The mimics of zeolites properties associated with MOFs characteristic gives them specific features consisting tailorable porosity, high inner surface area, ultrahigh thermal stability, and chemical robustness in a variety of organic solvents which led ZIFs as well-candidate materials for abundant functionalities in different majors as diverse as environmental engineering to the biomedical engineering field. The ZIFs could synthesize with hierarchical porous crystalline structure (mesoporous or microporous) so that they possess various morphological structures, crystalline patterns, and properties [48,49].

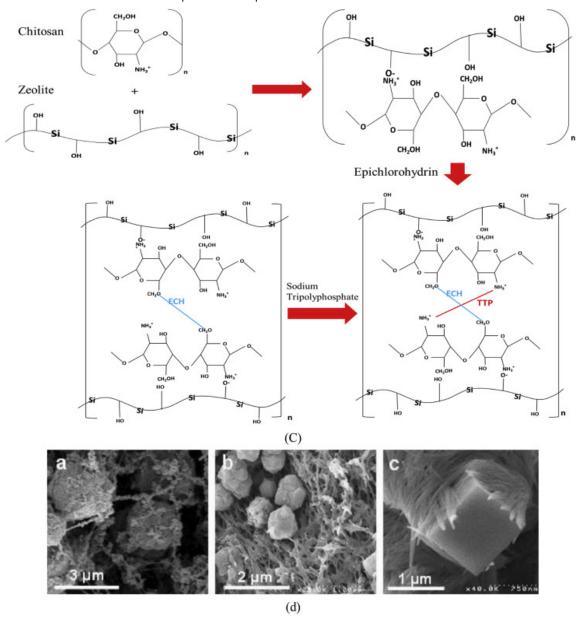
Chitosan is one of the outmatched derivatives of chitin with a broad spectrum of molecular weight from 1000 Da to  $2*10^5$  Da which can be found in the exoskeleton of crabs and shrimps, skeleton structure of many animals, and fungi cell walls. The low viscosity and better water solubility of chitosan with a molecular weight below the 10000 Da made it an excellent applicant for tissue engineering and biomedical demands [50]. Chitosan is a subset of polysaccharides including indiscriminately spread  $\beta$ -(1  $\rightarrow$  4)-linked p-glucosamine and N-acetyl-p-glucosamine with two functional groups (—NH2 and —OH) as active sites which made it applicable as excellent supporting bed and adsorbent for up taking diverse kind of polar pollutants and drug molecules (see Fig. 1). The amount of mole fraction of p-glucosamine in chitosan structure is elevated than chitin [51]. The general way to chitosan synthesis from chitin is the chemical treatment in an alkaline environment with concentrated sodium hydroxide in an aqueous solution which is called the deacetylation process (see Fig. 1). In the deacetylation process, —NH2 replaces the acetyl group in the main chain of chitin. Three major operational parameters including, reaction Time, NaOH concentration, and solution temperature effects on the purity, activity, and chitosan characteristics [52]. Changing —NH2 to —NH3+ through the protonation process at pH > 6.5 leads to an increase in the water-solubility of chitosan and its adhesive properties to negatively charged surfaces [53]. In addition, chitosan exhibits wide applications in the various fields from air/water purification to biomedical engineering due to its distinctive characteristics such as nontoxicity, eco-friendly, flexibility, and biodegradability.





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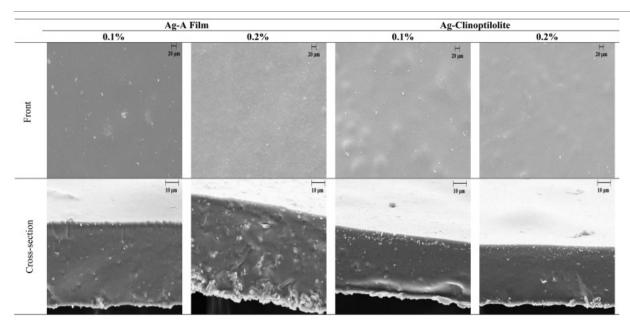


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Fig. 1. (A) Industrially most important zeolite structures including (a) Zeolite-A (b) Zeolite (X and Y), and (c) ZSM-5 (MFI topology) [160] (B) Chitin and chitosan structure, (a) N-Deacetylation of chitin to chitosan, (b) Schematic of chitosan molecule structure. (C) Proposed equations for the formation of chitosan–zeolite composites and also the structure of chitosan–zeolite composites (d) SEM images for (a) zeolite-X encapsulated in chitosan gel, (b) encapsulated zeolite-Y, (c) in-situ synthesized zeolite-A crystal [161].

Immersing as-synthesized zeolite particles into the chitosan-gelling solution and also the one-spot fabrication of zeolite frameworks into gel solution of chitosan hydrogel solution, are listed as the most popular way to produce various kinds of zeolite/chitosan hybrid composites (see Fig. 1). Needless to say, the hybrid combination of chitosan and zeolite crystals elevated the properties of both materials at the same time and makes a more practical material for more sophisticated applications. Also, it is proven that the existence of zeolite particles in the acidic solution of chitosan gel is well stable and the characteristics of zeolite-bearing chitosan xerogels including specific surface area and the pore volume are comparable to the of chitosan aerogels (120).

Typically, the type of zeolite and the applied dosage play a pivotal role in the properties of the zeolite/chitosan complex. Barbosa et al. [54] have performed a comprehensive study on the impact of various fractions of clinoptilolite and 4A-type zeolites in the chitosan film matrix. Using a homogenizer, it was tried to disperse different fractions of zeolite particles in the matrix of chitosan solution. The membranes were subsequently provided by casting solution method. Front SEM images of the pristine chitosan (2%) film and the composite indicated a smooth and homogeneous microstructure morphology and similar topology. Nevertheless, existing different types of zeolites with a varied extent in the membrane matrix made cross-sectional images a little different. These images indicated that in the membrane with a higher content of zeolites, particles tend to settle down in the lower surface of the membrane (Fig. 2.). Moreover, their findings showed that as the fraction of zeolites in the composite structure increased up to 2%, water vapor permeation decreased and higher thermal stability was achieved. However, as the zeolite fraction surpassed 2%, it adversely affected the mechanical stability of the composite toward the films tend to be more fragile and brittle.



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Fig. 2. SEM images of zeolite/chitosan membrane with 0.1 an 0.2% fraction of zeolite (a) Surface images of film (b) cross-sectional images.

# 3. Applications of zeolite-chitosan composite

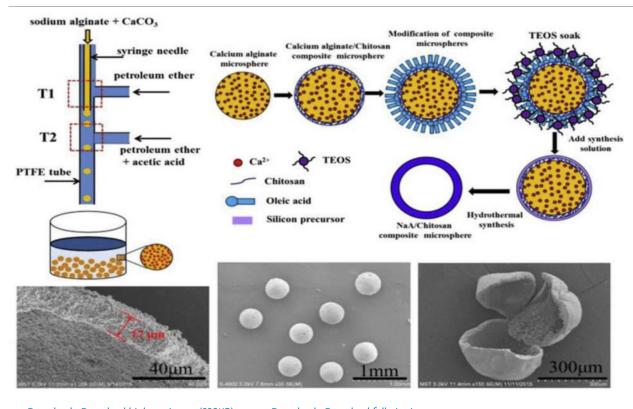
## 3.1. Environmental engineering applications

#### 3.1.1. Adsorption

The adsorption process of different organic/inorganic contaminations from aqueous and gas phase is one of the most traditional and practical techniques [55,56]. Designing and utilization of low-cost and high porous complexes as adsorbents or supporting bed with high removal ability are of utmost importance [[57], [58], [59]]. Zeolite has been widely utilized as an adsorption substrate that can be used in separation, drying and purification processes [60]. Zeolite and its composites have been used for dehydration, gas separation and molecule screening. Zeolite performance depends on pore size and the number of cations around the pore [61,62].

Lu et al. fabricated a strong adsorbent by optimizing the weight fraction of zeolite particles in the zeolite/chitosan complex. They found the composite with 60 wt% zeolite showed better copper cations adsorption ability in comparison to complexes with 50 wt% and 70 wt%, pure zeolite, and chitosan. In the acidic region, with a reduction in pH (—NH2) in chitosan alter to (—NH3+) and decrease the copper cations adsorption [61]. Ngah et al. [63,64] studied characteristics and Cu(II) re FEEDBACK  $\bigcirc$ 

three different chitosan/zeolite complexes. According to the outcomes of their study, the adsorption capacity of chitosan/zeolite crosslinked with epichlorohydrin and sodium tripolyphosphate (51.32 mg/g) was higher than that of the uncrosslinked chitosan/zeolite (25.61 mg/g) and chitosan/zeolite crosslinked with epichlorohydrin (14.75 mg/g). The (-CH<sub>2</sub>OH) groups in the chitosan structure play a key role in the adsorption mechanism of Cu(II). In a similar work by Ngah et al. [65], they applied uncrosslinked chitosan/zeolite for the elimination of copper cations. The maximum adsorption capacity of the adsorbent at pH 3 was reported 25.88 mg/g. The isotherm and kinetic study revealed that the adsorption process followed Langmuir and firstorder kinetic models, respectively. Moreover, the regeneration of the adsorbent was performed by EDTA. Mukhopadhyaya et al. synthesized zeolite/chitosan nanocomposite using the phase inversion method for arsenic removal from water. It was revealed that the zeolite content increase in nanocomposite was enhanced in hydrophilicity, the flux of membrane and porosity [66]. Various types of zeolite such as NaY, NaP, L, Phillipsite-Na, Z, and Fujasite-Na along with orthoclase mineral can be produced by altering the reaction factors like temperature, synthesis time, Si/Al ratio and crystallization template. It was reported that the zeolite NaY/chitosan was used to remove lead (III) from an aqueous solution. Adsorption data were fitted to the Langmuir isotherm model and kinetic studies confirmed that the lead (III) removal obeyed the second-order model [67]. Yan et al. synthesized hollow zeolite/chitosan particle via hydrolysis-gelation-hydrothermal technique. As shown in Fig. 3, the template was fabricated based on alginate microparticles coated with chitosan. After that, microparticles were treated by elainic acid and tetraethylorthosilicate (TEOS). Meantime the hydrothermal process, TEOS was hydrolyzed and it formed the shell of particles and alginate was removed during alkaline synthesis and the hollow core was formed. Such a platform can be used in adsorption of various ions like Cu<sup>+2</sup> [68].



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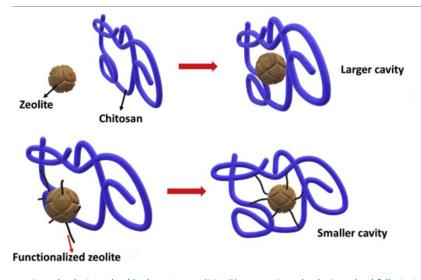
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Fig. 3. The synthesis procedure of the hollow zeolite/chitosan particle.

Kusrini et al. modified clinoptilolite zeolites with chitosan and iron oxide to remove the mercury from the liquid condensate hydrocarbon. Chitosan-modified zeolite enhanced the mercury rejection rate from 4.5 to 35% compared to the pristine zeolite. Moreover, iron oxide modification enhanced the removal percentage of mercury to 65%. It was hypothesized that the chitosan modifications enhanced both the functional groups and the removal efficiency. In addition, another hypothesis is that the iron oxide nanoparticles enhanced the surface area and zeolite/chitosan/iron oxide exhibited the synergic effect and dramatically increased the removal of mercury [69]. The functional groups of Chitosan react with various materials such as fit precedence of the pristine zeolite.

known as a pollutant and threat to human health. However, inferior mechanical property hinders chitosan usage in fluorine removal. Orozco et al. found that in situ syntheses of zeolite/chitosan composite led to the formation of the chemical interaction between chitosan and zeolite components, which resulted in better adsorption than physical mixing. Higher content of chitosan resulted in pore constriction and reduced the adsorption, thus the chitosan content was optimized [70]. It was reported that the zeolite/chitosan composite is a cost-effective and efficient substrate for dye removals such as reactive red and methylene blue [71], [72], [73]]. Zeolite/chitosan composite can interact with various pollutants due to both the positive charge of the chitosan and the negative charge of the zeolite. Xei et al. [74] utilized such composite to remove cationic (ammonium), anionic (phosphate) and organic (humic acid) pollutants from water. Adsorption ability of chitosan/zeolite and surfactant-modified chitosan/zeolite was comprehensively assessed on humic acid (HA) elimination in pH range from 4 to 12 by Lin and Zhan [75]. According to their founding, with an increase in solution pH, the removal efficiency of HA decreased. However, with an increase in adsorbent dose, contact time, and initial concentration of HA the removal percentage was enhanced. Moreover, they report an increase in the solution temperature directly influences the elimination efficiency of HA due to the endothermic nature of the adsorption process. Furthermore, the isotherm study exhibited the Langmuir model better fitted with the experiential data. In alkaline conditions, due to the deprotonation of amino groups in chitosan architecture resulting in weakening the electrostatic forces between adsorbent and HA molecules, the desorption of HA was taken place up to 66.6%.

Wafiroh et al. synthesized phosphorylated zeolite/chitosan as a proton exchange membrane fuel cell based on its proper mechanical properties, proton conductivity, swelling properties, thermal resistance, and methanol permeability [76]. Wang et al. [77] studied the influence of adding zeolite beta particles with diverse sizes and shapes in the chitosan membrane matrix for application in the direct methanol fuel cell. The presence of zeolite particles in the chitosan matrix decreased the permeability of methanol through the membrane due to their optimum free volume and methanol diffusion features. Moreover, modified zeolite particles by sulfonic group exhibited a higher ability to reduce methanol permeability. Interactions between modified zeolite particles and chitosan were considered as the main reason for the lower permeability of the complex. In a similar report, Wu et al. [78] applied modified NaY zeolite with aminopropyl groups or sulfonic propyl groups as filler into the chitosan membrane matrix to reduce methanol permeability for direct methanol fuel cell (Fig. 4). The transitional phase generated between chitosan structure and zeolite particles was recognized as the main cause of the reduction in methanol permeation. The complex which possesses modified zeolite with sulfonic propyl groups shows higher resistance to methanol permeation compared to complex possesses modified zeolite with aminopropyl groups.



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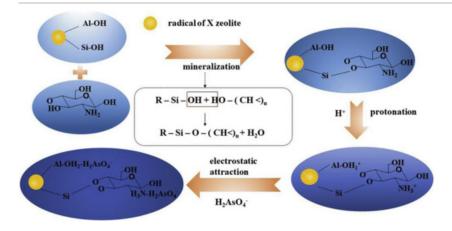
Fig. 4. Schematic illustration of the zeolite/chitosan membrane and modified-zeolite/chitosan membrane.

Yuan et al. [79] used sorbitol-plasticized chitosan/mordenite membrane for water/methanol adsorption and methanol permeability then proposed a correlation between interfacial morphology of membrane and methanol permeability. The presence of mordenite not only filled the voids in the chitosan structure but also enhanced the rigidity and physical properties of mordenite not only filled the voids in the chitosan structure but also enhanced the rigidity and physical properties of mordenite not only filled the voids in the chitosan structure but also enhanced the rigidity and physical properties of mordenite not only filled the voids in the chitosan structure but also enhanced the rigidity and physical properties of mordenite not only filled the voids in the chitosan structure but also enhanced the rigidity and physical properties of mordenite not only filled the voids in the chitosan structure but also enhanced the rigidity and physical properties of mordenite not only filled the voids in the chitosan structure but also enhanced the rigidity and physical properties of mordenite not only filled the voids in the chitosan structure but also enhanced the rigidity and physical properties of the properties of

stability of the complex. Methanol permeability by the fabricated complex was 44% and 400% lower than the pristine chitosan membrane and Nafion, respectively.

Wang et al. [80] added various zeolite (e.g., 3A, 4A, 5A, 13X, mordenite, and HZSM-5) with different silicon/aluminum ratio to the chitosan membrane matrix and studied their ability in methanol permeation. According to their observation, with an increase in silicon/aluminum ratio, the hydrogen-bonding between zeolite particles and chitosan molecules become stronger and make cavities smaller. Zeolites with higher silicon/aluminum ratio exhibited higher methanol adsorption resulting in lower swelling. Furthermore, methanol permeability was reduced by an increase in the zeolite particle size.

Han et al. synthesized chitosan-NaX zeolite from fly ash to remove arsenic from wastewater. The mechanism of arsenic removal is depicted in Fig. 5. It was proposed that the Si–OH groups on NaX Zeolite can be mineralized by C–OH groups on chitosan to generate the interpenetrating network, which is the process that fixed chitosan within the zeolite structure. Al–OH and NH<sub>2</sub> groups belong to composite were protonated in the acidic region and the surface charge of the platform became positive which then interacted with arsenic [81].



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Fig. 5. The schematic mechanism for arsenic adsorption using zeolite/chitosan composite.

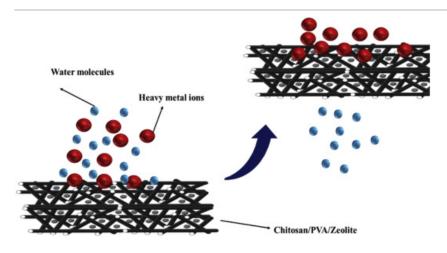
#### 3.1.1.1. Adsorption mechanism

Zeolites, as a whole, have been very popular for the separation of various gases. There have been different possible mechanisms involved in gas separation for different zeolites. Shang et al. exhibited a unique separating mechanism for CO and N<sub>2</sub> separation for chabazite zeolite. Contrary to molecular sieving, it was shown that a larger CO molecule was in preference to a small N2 molecule. The separation mechanism was based on the difference of ability of a guest molecule to induce temporary and reversible cation deviation from the center of pore aperture. The separating mechanism was similar to the molecular trapdoor [82]. Montanari et al. did a comparative study for CO<sub>2</sub> adsorption on zeolites 3A (K-LTA), 4A (Na-LTA), and 3A (Ca,Na-LTA). For 3A zeolite, CO<sub>2</sub> adsorption was mostly limited to the external surface. However, for 4A and 5A zeolites the adsorption of both types of molecules and carbonate species occur mostly in cavities. The most of carbonate-like species are fabricated on 4A than on 5A zeolite. This existing gap is induced by the partial poisoning of 5A cations [83]. Khalid et al. used zeolites to remove phenol from aqueous solution and its adsorption properties were compared with activated carbon. Zeolites with a high Si/Al ratio were hydrophobic which were suitable for phenol adsorption. BEA zeolite was used successfully and the adsorption capacity was higher at low phenol concentration [84]. Recent advances in natural/synthetic zeolites for CO2 capture have been fully recorded by Bonenfant et al. report. Incorporation of various cations into channels of zeolite structure leads to basicity and an electric field governs the CO<sub>2</sub> adsorption. The basicity of the electric field varies significantly with Si/Al ratio. It was also exhibited that an increase in pressure value, as well as a decrease in temperature, has a positive effect on the CO<sub>2</sub> adsorption amount [85]. Langmi et al. investigated the use of zeolites for storing hydrogen storage material. Different zeolites like A, X, H and RHO were synthesized with hydrothermal methods. Phase composition and crystallinity were checked with XRD. Results displayed that hydrogen uptake in zeolites is dependent on temperature, framework and cation type [86]. Janche FEEDBACK ♥ investigated the sorption properties of modified zeolites and mesoporous materials against the water with physio-chemical methods. Selected materials were pelleted and investigated in lab-scaled storage to prove the performance of modified storage materials. Impregnated mesoporous materials show a much lower temperature lift than zeolite [87].

#### 3.1.2. Membrane separation

Contamination separation from water resources and air through the different process of membrane methodology such as ion exchange, reverse osmosis and nanofiltration has grabbed a substantial deal of attention due to low cost, ease of operation and safety issues. However, some most serious disadvantageous of membrane including non-biodegradability and low mechanical properties restricted their application in harsh conditions. Characteristics of a membrane are chiefly influenced by the size, porosity, and functional groups of composed materials and the surface morphology of the membrane. Controlling the characteristics of composed materials leads to the design of a membrane with high adsorption capacity, low swelling rate, and adequate hydrophilicity.

To address the nonbiodegradability of membranes, scientists have tried to apply biopolymers like chitosan to fabricate nontoxic and biocompatible frameworks that are safer and do less damage to the environment. Habiba et al. [88] successfully synthesized a uniform nanofibrous membrane with high chemical/physical stability in pure water, high acidic, and basic environment using electrospinning of ternary mixture of hydrolyzed-chitosan/PVA/zeolite with optimized PVA to chitosan ratio (50:50) and 0.1 wt% zeolite particles. Various explanations were reported to justify the stability of membrane in different pH environments like chitosan insolubility and hydrogen bonding interconnections among the materials. However, decreases in internal pore sizes of membrane reported as the possible reason for small shrinkage of the membrane in the acidic region. The resulting membrane exhibits high adsorption kinetic and fair adsorption capacity at low contamination concentrations of three hazardous metal ions (Ni (II), Ferric, and chromium (VI)). This eco-friendly, biodegradable membrane showed high stability and adsorption efficiency after five repeated removal process, so that proved itself as a feasible and practical membrane for real-word demands (see Fig. 6). In another similar report, Habiba et al. applied this membrane for a ternary pollutant system including Congo red, methyl orange, and chromium (VI). Removal of Congo red and methyl orange followed the flocculation with high efficiency at a dye concentration of more than 100 mg/L. For methyl orange below the 100 mg/L and for Chromium (VI) at all concentrations the separation took place through the adsorption process [89].



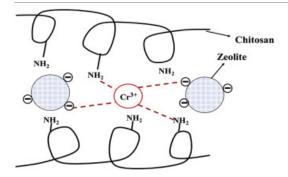
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Fig. 6. Schematic of heavy metal ion separation by chitosan/PVA/zeolite membrane.

In another report, Batista et al. [90] enhanced the physical stability of chitosan film by crosslinking and combining with zeolite particles then employed it for Cr(VI) elimination from the aqueous environment. According to their work, pristine chitosan film dissolved quickly at the higher acidic environment (pH 4), however adding zeolite into the chitosan matrix increased its stability. The results of their observation demonstrate that the complex had higher thermal stability and maximum adsorption capacity (17.28 mg/g) compared to pristine chitosan film.

Truong et al. [91] attempted to optimized the weight fraction of zeolite in the chitosan-based membrane which supported glutaraldehyde crosslinking. The best composite was synthesized with 20 wt% zeolite and applied to removal of four common, toxic heavy metals (Cr, As, Cd and Pb) by the evacuation permeation process (see Fig. 7). Their observation showed that the Cr selectivity of the membrane due to electrostatic forces between the amino group of chitosan and induced-negative charge on zeolite particles was at its climax when the pH solution was 5.5. The negative charge of the zeolite surface at acidic region due to the presence of the amino group in its structure leads to the uptake of cationic metal ions and the permeability factor of the membrane was higher for Cr ions in comparison to AS. The recycling test was performed to survey the reusability ability of the membrane. The reported results showed the reduction of membrane adsorption capacity after several cycles were less than 10% of its initial state.



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Fig. 7. Mechanism of Cr<sup>3+</sup> separation process by chitosan/zeolite membrane.

Mukhopadhyaya et al. [66] synthesized zeolite/chitosan nanocomposite using the phase inversion technique for arsenic removal from water. They found the presence of zeolite increased the porosity of membrane more than two times. In addition, the hydrophilicity of membrane remarkably elevated and it's proven by a reduction in water contact angle. The optimization of zeolite content in the membrane complex, electrostatic interaction between zeolite and pollutant molecules, increment in the porosity, and wettability of membrane increased the Arsenic rejection up to more than 94% at high concentration of Arsenic cations [66].

Nawawi et al. modified the chitosan membrane by adding different amounts of zeolite-A and treated with an alkaline solution for the application in the pervaporation separation of the isopropanol-water mixture. The synthesized membrane showed low swelling and high mechanical properties. In addition, Their findings depicted that Zeolite-A/chitosan with a ratio of 1:8 had a superior performance for isopropanol-water mixture separation [92].

#### 3.1.3. Catalyst

Zeolites have been utilized as catalysts for various reactions such as isomerization, hydrocarbon synthesis, dehydrogenation and cracking, in which zeolite can ameliorate the various reactions such as metal-induced and acid-base reactions. Zeolites act as a shape-selective oxidation catalyst based on transition-state selectivity and molecule size, in which the reaction of absorbed molecules occurs within the zeolite pores to achieve the high level of product control [[93], [94], [95], [96]]. Junhua et al. [97] performed removal of NO<sub>x</sub> by catalytic technology at low temperature (100–300 °C) with metal exchanged zeolite catalyst. The impact of catalyst preparation method, precursor, and various supports was investigated. It was reported that chitosan amine groups enhanced zeolite activity and promote catalytic efficiency [98]. Wang et al. [99] used chitosan film to control the orientation of TS-1 crystals on a porous alumina support. The crystal grains which were formed were b-oriented regarding the pole figure and crystalline structural analysis. The produced membrane exhibited impressive selectivity for the oxidation-catalysis experiment [99]. Zhang et al. prepared zeolite/chitosan (Zel/Chi) monoliths through a unidirectional freeze-drying technique. The uptake of metal ions and also the drug release efficiency of the Zel/Chi monoliths with the porous structure were examined and reported. For Cu<sup>+2</sup> capture, the maximum adsorption value was recorded 89 mg/g. The Cu<sup>+2</sup>-adsorbed Zel/Chi monoliths, as a recyclable catalyst, showed excellent performance in the reduction of 4-nitrophenol to 4-aminophenol [62]. Lin et

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al, used hydrothermal synthetic method to prepare mesoporous zeolite ZSM-5 crystals by using ammonium-modified chitosan and tetrapropylammonium hydroxide (TPOAH) as the meso-and micro-scale template. As prepared mesoporous ZSM-5 exhibited enhanced catalytic activity compared to conventional ZSM-5 for Claisen-Schmidt condensation reaction of 2hydroxylacetophenone [100]. Chen et al. prepared a novel zeolite (HY)-filled polymeric chitosan membrane. The membrane was used for pervaporation experiments for the ethanol-water system. An increase in zeolite content caused the damage of the crystalline structure of chitosan, indicating a strong interaction between HY zeolite and chitosan. The highest separation factor was obtained for 20 mass% of HY zeolite content and remained constant during the PV process [101]. Yu et al. prepared homogenous microspherical-shaped zeolite-A/chitosan which possesses the elevated ability of water uptake (290 mg/g) due to its effective specific surface area (30 m<sup>2</sup>/g). The separation factor of 12 for the CH<sub>4</sub>/H<sub>2</sub> mixture was also obtained [102]. Kumar et al. synthesized zeolite/chitosan mesoporous composite for enhanced capture and catalytic activity in chemical fixation of CO<sub>2</sub> in environmental applications. Zeolite/chitosan composite exhibited a synergistic effect and great catalytic performance in CO2 fixation within cyclic carbonates [34]. Morsli et al. synthesized the zeolite/chitosan composite using in situ synthesis and encapsulation methods. It was found that the in situ synthesis of zeolite/chitosan composite resulted in better adsorption and catalytic effect because more porosity was available [103]. Fereidooni et al. utilized zeolite/chitosan composite as a solid heterogeneous catalyst to produce biodiesel using trans-esterification of waste cooking oil to methyl esters. Waste cooking oil (WCO) was used as feedstock for biodiesel production. KOH-modification of natural zeolite leads to an increase in amount of silica content in its framework and raises the K+ content through hydroxylpotaslite formation as well. For alcohol/oil ratio of 1:7 at 40 V in presence of 2 wt% water, the yield of biodiesel from WCO was 93 wt% for 1 wt% catalyst [104]. It was demonstrated that the zeolite treatment with KOH reduced the silica concentration and enhanced the K<sup>+</sup> via hydroxylpotaslite formation [104].

# 3.2. Biomedical applications

Biomedical engineering is the translation of engineering and applied science concepts to medicine and biology for enhancing the level of health [[105], [106], [107], [108]]. Biomedical engineering endeavors to fill the gap between engineering/material science and medicine [109,110]. Tissue engineering, as a subgroup of biomedical engineering, is for designing appropriate substrates to enhance cellular activity and tissue regeneration [[111], [112], [113]]. In this regard, various materials have been tailored to exhibit maximum regeneration [[114], [115], [116]]. Cellular cytotoxicity evaluation of various types of zeolite reveals that the zeolites are biocompatible and safe for medical usage depending on size, shape, surface chemistry, and dosage [117]. Because of its low toxicity and tailorable structure, and because cages and channel form micrometer to the nanometer, zeolite can be used in various fields such as drug delivery, tissue engineering, and regenerative medicine [118,119]. Indeed, the addition of zeolites into the chitosan matrix can be expected to improve biomedical engineering characteristics of chitosan-based hydrogels.

Zeolite/chitosan composite application is summarized in Table 1 [[88,[120], [121], [122], [123], [124], [125], [126]]].

Table 1. Zeolite/chitosan frameworks and their applications.

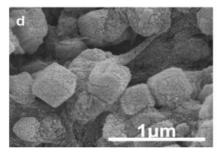
Material	Applications	Comments
Zeolite/chitosan	Removal of arsenic	Enhanced hydrophobicity thanks to the enlargement of the pore size of the membrane upon the addition of zeolite content.
$\label{lem:continuous} Zeolite/chitosan/bentonite/active \\ carbon$	Removal of cadmium	Enhanced soil pH and decreased biological effectiveness of heavy metals.
Chitosan-coated Na–X zeolite	Removal of arsenic	The removal of arsenic was significantly performed by the formation of bonds of As–N and As–O. Moreover, the surface hydroxyl group of Al–OH and $-{\rm NH_2}$ facilitated arsenic removal from wastewater.
Zeolite loaded in PLA/chitosan nanofiber	Drug release	Doxorubicin was loaded in zeolite and following the Fickian diffusion release mechanism.
zeolite-chitosan	Catalyst	Mesoporous substrate enabled for CO <sub>2</sub> fixation.

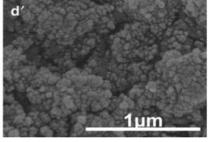
Material	Applications	Comments
Zeolite/Chitosan/polyvinyl alcohol	Adsorption of methyl orange	Nanofiber mechanical properties can be tuned by zeolite. adsorption mechanism obeys the Freundlich model. pH reduction enhanced adsorption.
Zeolite-loaded alginate/chitosan	Wound dressing	Hydrogel beads used as a topical hemostat. Promote blood coagulation via multiple mechanisms: erythrocyte adhesion, factor concentration, and the ability to serve as a mechanical barrier to blood loss.
Zeolite-chitosan	Wound dressing	Enhanced antioxidant and antimicrobial properties were achieved.
Zeolite-chitosan	Biological survey	Nanocomposite improved the histological structure of rainbow trout intestine compared to conventional composite.
Ag-zeolite-chitosan	antibacterial surface	The antibacterial agent was developed as a ceramic filler to endow the antibacterial feature to composite.
Fly ash-zeolite-chitosan	Absorption of heavy metal ions	Sorption efficiency with various ions $Cu\left(II\right)>Fe\left(III\right)>Zn\left(II\right)>Mn\left(II\right)$
Magnetic zeolite/chitosan/polyacrylonitrile	Absorption	Nanofiber-coated sponges facilitated water-oil separation for many cycles.
Zeolite/chitosan/PVA	Adsorption	Nanofiber removed Cr (VI), Fe (III), and Ni (II) ions from wastewater.

#### 3.2.1. Tissue engineering

Using various kinds of zeolites to promote the properties of chitosan and fabricate robust hybrid composites in order to stimulate the rehabilitation and regeneration of body tissues is a pretty new hot biomedical-research topic in recent years. Considering the specific features which high porous zeolite materials have given to zeolite/chitosan hybrid composites such as facile shape adjustment and excellent mechanical properties, Akmammedov et al. [127] prepared a freeze-dried scaffold based on zeolite-A/chitosan nanocomposite with various zeolite mass ratio as diverse as 0.5, 1.0, and 2.0 wt% and introduced it to assess viability, cell attachment, and Human Bone Marrow-derived Mesenchymal Stem Cells (hBMMSC) proliferation. The porous structure facilitated the nutrient and cytokine diffusion within the scaffold and waste products diffused out. It was shown that the zeolite-A/chitosan containing 0.5% of zeolite had higher efficiency in viability and attachments of hBMMSC and also it was better for cellular activity than pristine chitosan.

Yu et al. [127] through an in vitro assay using simulated body fluid (SBF), studied the impact of Ca<sup>2+</sup> exchanged zeolite-A weight fraction in a blend of chitosan matrix (in all cylinders, plates, and thin films forms) onto the rate of hydroxyapatite (HAP) formation. The results of their work indicate that using Ca<sup>2+</sup> exchanged zeolite-A/chitosan, increased the formation rate of HAP in comparison to zeolite type A/chitosan. This is mainly because, despite that the HAP only grows on the cubic-shaped crystals of zeolite-A, these nanoparticles were formed onto the whole Ca<sup>2+</sup> exchanged zeolite-A surface including its channels (see Fig. 8). Actually, an increase in the extent of calcium cations by increasing in the weigh fraction of Ca<sup>2+</sup> exchanged zeolite-A from 20% up to 55% in zeolite/chitosan bulk, improves the growth of HAP nanoparticles.





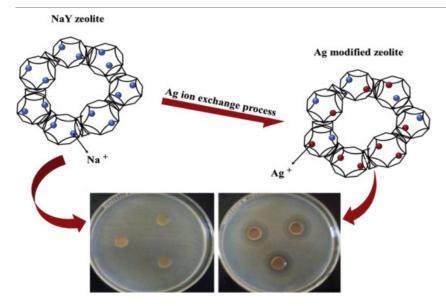
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Fig. 8. Scanning electronic morphology images of 55 wt% (d) and  $Ca^{2+}$  exchanged zeolite-A/chitosan (d') as-synthesized zeolite-A/chitosan hybrid composites.

#### 3.2.2. Antibacterial and wound healing dress

By the development of cosmetics and dermatology demands, the application of natural zeolites as an excellent UV protector and wound cure agents in biomedical industries is on the rise. Incorporation of silver metal into the framework of diverse types of natural zeolite to achieve silver-based agent, through a process called ion-exchange, have been aroused much interest among researchers. Taaca et al. [127] have designed a series of studies on the fabrication and characterization of Ag-zeolite/chitosan composite for antimicrobial uses (see Fig. 9). To improve the antibacterial activity of chitosan-based implants, AgZ nanoparticles, as ceramic fillers, were synthesized by incorporation of Ag into Philippine natural zeolite (NaZ). Then, it was introduced to the chitosan biopolymer matrix and the composite was examined against S. aureus as well as E. coli and. The outcomes revealed AgZ-chitosan composite could appreciably impede bacterial growth [128]. In another work, she tried to enhance the antibacterial activity of AgZ-chitosan agents through surface modification of it via plasma treatment. Indeed, Ag presence resulted in antibacterial activity of the composite, and plasma treatment elevated the wettability and adhesion of the zeolite/chitosan composite. It was proposed that the plasma-treated Ag-zeolite/chitosan composite can be used in tissue engineering, especially as a wound dressing [129,130]. A new hybrid composite was developed based on Ag<sup>+</sup> enriched zeolite-A/chitosan to play a role as a powerful agent for inactivation microbial growth. In this study, Yu and his colleges [131] tried to exchange silver elements with Na at sodalite cages of synthesized zeolite-A, which are connected by four-membered rings forming a uniform 3D microporous structure. Moreover, using the electrostatic force between the anionic surface of clinoptilolite microparticles and cationic tails of chitosan biopolymer, a chitosan-coated clinoptilolite composite was obtained by Tegl et al. [130]. Chemo-enzymatic modification of this hybrid composite with caffeic acid (CA) agent aided by laccase generating stable particles, as well as post-functionalized via glucose oxidase (GOX), leads to an immense improvement in its antibacterial activity. Scacchettia et al. evaluated the quality of a cotton fabric crosslinked by citric acid to the Ag-zeolite/chitosan matrix regarding antimicrobial functionalities whereas its air and water permeability had a negligible reduction [130].



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Fig. 9. Exchanging of Ag+ with Na+ in the framework of NaZ zeolite for antibacterial applications.

Chitosan has been vastly utilized for wound dressing due to its highly appealing results as the wound healing and blood stopper agent [132]. Using chitosan-based patches for hemostasis purposes, its amino groups attract the negatively charged red blood cells and subsequently form a scab at the addressed point. Scientists also believe that interacting with erythrocytes and/or alteration in platelets activity in the injury site, and also the interaction between polycationic characteristics of chitosan and nonspecific binding to cell membranes could be the possible mechanisms for controlling aggressive bleeding. On the other hand, from a zeolite point of view, many types of them have shown good hemorrhage control capability in the in vivo tests. According to the literature, it is considered that an exothermic water adoption phenomenon is the governing mechanism for the treatment of traumatic hemorrhage using zeolite particles [133].

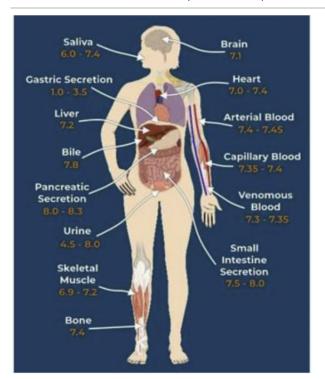
In such a way, zeolite-loaded chitosan composites have shown a synergistic impact toward higher hemostatic activity. Fathi and coworkers [122] designed a strategy to investigate the efficiency of zeolite-laden hydrogel for blood clotting applications. In this work, to encapsulate sodium zeolite particles in alginate/chitosan beads, firstly, they prepared a solution containing zeolite dispersed in alginate solution with a 1:1 or 2:1 w/v ration. Then, the prepared solution was dropped wisely added into a mixing mixture consist of chitosan, CaCl2, and acetic acid with 1% (w/v) of each component. Next, fabricated hybrid composites introduced to in vitro assessment to study its hemostatic capabilities. Live/dead assessment indicated that the alginate/chitosan coating has appreciably alleviated the cytotoxicity of zeolite particles on living cells. Also, the side effects of produced composite onto the damaged cells in the targeted point were decreased in comparison to the pristine zeolite. The authors have explained that chitosan excellent adhesion feature along with the adsorption of blood content by zeolite is the main reason for the good efficacy of zeolite/chitosan composites as a topical hemostat.

In another similar work, Huang et al. [134] studied the implementation of self-assembling hydrophobically modified chitosan (HCs)/ZSM-5 to stop minor and severe bleeding. The produced composite showed good biocompatibility since both HCs and ZSM-5 considered biocompatible material. Moreover, cytotoxicity assessment of materials validated negligible side effects on cell viability due to the excellent cell viability of pristine HCs and zeolite particles. According to the data from blood clotting (HCs)/ZSM-5 provided a facile and efficient hemostasis activity (almost  $86 \pm 5$  s) rather than both ingredients in pure form.

# 3.2.3. Drug delivery and cancer therapy

According to the latest news of the World Cancer Report, the population who are suffering from cancer will increase to 15 million cases by 2020, therefore, synthesis and applications of cost-efficient and potent anticancer drug delivery systems based on zeolite/chitosan carrier have become so important.

According to Fig. 10 different human body organs have various levels of pH (mostly alkaline environment). Moreover, different pathological spots (e.g., ischemia [135], infections [136], and inflammation [137]) shows different pH profile compared to real tissues. Therefore, it is of utmost significance to select carriers with high resistance to a wide range of undesired pH levels. In other words, pH-responsive carries such as chitosan have a higher ability to keep drugs molecules in their matrix and release a higher amount of medicine agents in the target location with a specific pH environment. For instance, many researchers applied chitosan-based complexes for the delivery of anticancer drug molecules like doxorubicin (DOX) to cancerous cells [138,139]. Cancerous spots generally have an acidic extracellular environment around 6–7 which is mostly because of their high proliferation rate and activities results in proton generation [139]. Accordingly, it is very important that a small amount of drug released from carries in the blood curriculum with a narrow pH window (~7.3–7.5) in its way to the target tumor cells. Although there is a lot of studies on drug delivery by chitosan-based materials, a limited publication has been reported on chitosan/zeolite compositions for delivery purposes [123,140,141].

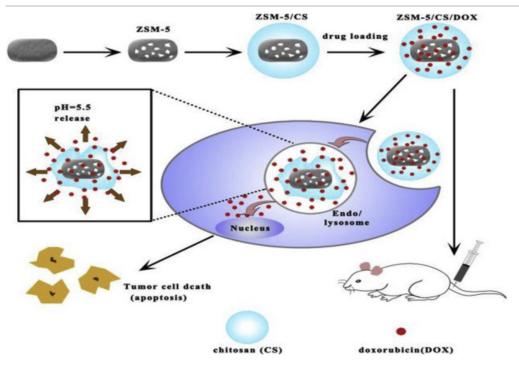


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Fig. 10. The pH of various organs in the human body [162].

Synthesis of different platforms based on ZSM-5 zeolite and chitosan combination due to its synergistic characters, as a robust anticancer, has attracted so many interests among researchers. Zeolite/chitosan was used in intravenous chemotherapy in which DOX was loaded in mesoporous zeolite/chitosan and injected in a rat. The acidic environment of cancer resulted in chitosan dissolution and the anticancer drug was released. Wen et al. fabricated ZSM-5 zeolite/chitosan via desilication (hollow core and mesoporous shell) and loaded it with DOX as a pH-sensitive delivery system to treat osteosarcoma. The existence of lysosome and endosome in the tumor caused the milieu to be acidic and chitosan was dissolved in acidic media and the drug was released in the tumor area. Fan and the colleges [142] also designed a pH-sensitive DOX carrier by the synthesis of a nanodisk-shaped mesoporous ZSM-5 zeolite/chitosan hybrid with low cardiac toxicity. The outcomes validated that in the acidic region like tumor vicinity, the composite showed high efficacy against osteosarcoma because of its quick rate of DOX release (see Fig. 11).



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Fig. 11. Illustration of ZSM-5 zeolite/chitosan composite as a DOX carrier for tumor treatment.

Abasian et al. [143] loaded doxorubicin into the magnetic NaX zeolite and embedded it within the PLA/chitosan nanofibers to control the drug release rate, which obeyed the Fickian diffusion; such nanocomposite exhibited antitumor activity, demonstrated by carcinoma cell death. In another report by Zhao et al. [144], zeolite encapsulated by chitosan-crosslinked TiO2 spherical-shaped composite was utilized as a supporting bed to green fabricate the different size ranges of selenium metalloid nanoparticles (Se NPs) through self-assembly and UV-light aided process onto molecular-imprinted sites located on its surface. According to the unique characteristics of nanosized Se spheres, they showed a quick and wonderful efficiency in diagnostic detection of small cell lung cancer biomarkers.

Tiburu et al. [145] synthesized the zeolite/chitosan nanoparticle via ionic crosslinking using pentasodium triphosphate as an anticancer platform. To study the possible cytotoxicity activity of such nanocomposite, it incubated with cancer cells (HeLa), and it was observed that the HeLa cells' growth was appreciably decreased. Tegl et al. [123] found out using a natural phenol crosslinker such as caffeic acid to post-modify chitosan-coated clinoptilolite composite in order to immobilization of glucose oxidase (GOX) for antibacterial goals, gives it considerable antioxidant properties.

#### 3.3. Other potential applications

So far, despite environmental and biomedical engineering fields as the most significant focal point of zeolite/chitosan composites functionalities, other potential applications have been introduced. Wafiroh et al. [146] synthesized phosphorylated zeolite-A/chitosan as a proton exchange membrane fuel cell based on its proper mechanical properties, proton conductivity (3.2  $10^{-4}$  S/cm), swelling properties, thermal resistance up to 415 K, and methanol permeability. The chitosan used in this study was extracted and purified from shrimp shell wastes. To reduce the swelling of chitosan in water, the crosslinking technique was performed. The combination of low cost, the biocompatible natural polymer in this complex made it applicable as an environmental-friendly electrolyte membrane for fuel cell demands.

Likewise, Wang et al. applied various hydrophilic/hydrophobic zeolites in the framework of chitosan to fabricate desired zeolite/chitosan complexes for application in direct methanol fuel cell. They reported that reduction in the diffusion resistance of methanol by adding hydrophilic zeolite to chitosan leads to the increment of the methanol permeability and free volume cavity size. Existence of a large number of water molecules, ( $-SO_4^{2-}$ ), and ( $-NH_3^+$ ) in bulk of cross-linked chitosar FEEDBACK  $\bigcirc$ 

protons transportation. Despite hydrophobic zeolites, the utilization of hydrophilic zeolite enhanced proton conductivity [123]. García et al. [147] used zeolite/chitosan film as protection cover on tomato during refrigerated storage. They assessed the efficiency of protection by measuring fungal decay, speed of respiration, quality and general visual appearance characteristics. The mentioned cover exhibited acceptable performance in delaying the ripening of tomatoes but could not prevent weight loss of tomatoes during the experiment.

# 4. Commercialization and challenges

The extraction of chitosan from shrimp's shell wastes and other low-cost resources as well as applying natural zeolite leads to a sharp rise in the utilization of biodegradable, nontoxic zeolite/chitosan complexes in recent years in many fields. Fortunately, besides existing a vast resource of natural zeolite in most regions of the world, nowadays, by commercialization various zeolite synthesis, as well as the industrialization of chitosan production from chitin origin with different deacetylation percentage, jointly pave the way of commercialization of zeolite/chitosan hybrid composites. Although these compositions were applied in various fields and showed excellent efficiency, many factors must be noticed before large-scale production. For instance, the optimum amount of zeolite in the composites, as well as hydrophilic or hydrophobic properties of zeolite, affect the characteristics of the complex. In addition, the cost/performance, quality, and how to uniform synthesis of material are the most parameters that should be figured out before mass production. Since these composites have not designed or formulated with specific purposes or formulation until now, there are always possibilities of discrepancies between the applications and prepared complexes and this is the most conspicuous reason that inhibits the existence of these products on the global market at least for now.

#### 5. Future perspective and concluding remarks

Altering the porosity form macroporous to mesoporous and microporous can be a game-changer in high-technology usages. Materials with controlled porous dispersity on the nano-scale play an essential role in various applications such as catalysis, drug delivery systems, tissue engineering, coatings, cosmetics, bio-separation, diagnostics, gas-separation, and nanotechnology. Zeolites possess an inherent limit on pore size and accessibility owing to the pore templates available for their synthesis. However, macroporous materials with a wide range of pores diameter from 50 to 1000 nm such as porous carbohydrates result in facile access to the internal pores at the cost of selectivity. Such shortcomings inspire scientist to develop the mesoporous materials which have an intermediate and controllable pore size range. Chitosan/zeolite as a biocompatible mesoporous material possesses narrow pore size distributions and high surface areas that exhibit the appropriate biocompatibility and low toxicity. Environmental concerns have grown in recent years and industries endeavor to utilize bio-based and biocompatible materials. Zeolite/chitosan composite can be attractive as a natural and cost-effective and properties-controlled material. The tunable structure of zeolite/chitosan (zeolite pores diameter and around ions) enhances its usage in various applications such as adsorption, catalyst and biomedical. For instance, zeolite can be used as a packaging film because of the proper adsorption of ethylene gas [147]. Moreover, zeolite composites can be utilized as polymerization substrate of conductive polymers, which yields unusual conductivity and can make frameworks in supercapacitors [148]. By altering the zeolite/chitosan properties, such composite can be used in various applications with desired and tunable properties [149]. For instance, adding modified nanoparticles like zeolite [[150], [151], [152]] and different polymers to the matrix of various substrates could improve their antifouling and abilities to selective filtration/adsorption [[153], [154], [155], [156], [157], [158], [159]]. Physical and chemical characteristics of various zeolite can be carefully modified to select specific molecules for separation technology uses. However, according to the best knowledge of the authors, there is no study on chitosan/zeolite complexes in these applications. Therefore, it is an open window to complex various zeolite types and their functionalized particles with chitosan and surveys their ability in the antifouling and selective membrane.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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# Appendix A. Supplementary data

The following is the Supplementary data to this article:

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Multimedia component 1.

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Research data for this article

- Data not available / No data was used for the research described in the article
- (i) About research data 7

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