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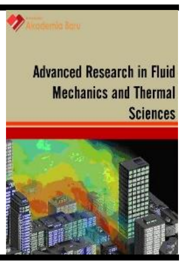

A Review on Thin Layered Composite Zeolite for Water Desalination Applications

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ABSTRACT

Seawater is one of the abundant water resources which is estimated covers about 71% of the earth's surface which is existed either in the form of oceans and seas. Thus, gaining freshwater from seawater is one of the best alternatives by considering of serious problem of water contamination in our mainstream system. In the water treatment, reverse osmosis (RO) pre-treatment, chemical, and biotechnology industries, ultrafiltration membrane have been used extensively for separation processes. Desalination is one of the processes that eliminates mineral content found in saline water via water treatment process. Various approaches used in desalination include adsorption, ion exchange, distillation, evaporation, reverse osmosis and electrodialysis. Even with all of the existing techniques, researchers are still exploring new method that are safe, energy-efficient and simple to operate. Therefore, the use of adsorption technique is still applicable until now. Currently, scientists are focusing on providing new and effective ways of desalination, removing anions and cations by using superpower particle of zeolites. This review paper aims to compile studies done by researchers on the effect of various kaolin towards a hydrothermal synthesis of zeolite. Adsorption performance of zeolite towards desalination is also included together with its mathematical model for more understanding on its performance.

1. Introduction

Although there are various techniques of water treatment, adsorption offers a simple, low-cost system with minimum energy consumption. Adsorption is one of technique that can be employed to remove salt and mineral from saline water. In fact, the adsorption technique has been used previously until now, since this approach provides a simple removal technique with high adsorption

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efficiency. It has the ability to eliminate a broad range of compounds which is convenient for industrial wastewater as it has various mineral content. Adsorption is commonly utilized in removal of non-degradable organic compounds having low concentrations from water sources and in drinking water processing. In adsorption, molecules from the liquid (adsorbate) will bind itself to the surface of solid substance (adsorbent). Thus, good adsorbent material should have a porous structure with an insoluble property that able to provide coated contaminant surface, such as capillaries and pores. Adsorbents with high surface area, either internal or outer layer will permit better adsorption. Few well-known good adsorbents include activated carbon, charcoal, silica gel and alumina. Among the listed adsorbents, activated carbons are the most favorable in removal of polar compound. Basically, lot of researchers has employed adsorption method in their studies to efficiently remove heavy metals from water bodies and waste streams [1–6]. In recent years, potential of zeolites as adsorbents were explored. Ayodeji *et al.*, [7] in his experimental work has successfully synthesized zeolites via hydrothermal method. He described that the technique requires mainly silica, alumina, metal cations, water and phosphorus which were then converted by alkaline supersaturated solution into zeolites with micropores crystal-like structure [8–10]. Natural zeolites or synthetic zeolites composed of alumina-silicate-polymer was discovered to possess a polar bonding sites with homogenous pores distribution which can allows a wide range adsorption application such as drying, purification and separation. In addition, mesoporous structure owned by zeolite provide large surface area for adsorption and thus, have a higher selectivity compared to active carbon.

Caputo and Pepe [11] reported that standard testing procedure for adsorption was usually done in either column or batch process. If it is a small-scale testing, batch process was utilized as it consumed less time. To understand mechanism of adsorption and performance of adsorption, equilibrium model named Freundlich and Langmuir isotherms were applied [12,13]. For design optimization, one need to find the suitable correlation that fits the equilibrium curves [14]. Next, column testing method are useful for remediation of larger scale wastewater and for cyclic adsorption-desorption process. Design of packed bed adsorber used for column testing was designed according to calculation of breakthrough curves obtained in small scale adsorption [15,16], where the mathematical analysis method was described in past existing literatures [17–20]. In the present work, adsorbent materials, including zeolite for water desalination, will be critically reviewed. According to previous works, soil, clay, mineral including zeolite have been used for ages in the water treatment process. Natural zeolite act as a good adsorbent, thus, it gives advantages in removing impurities at relatively low concentration. In this work, zeolite with controlled hydrothermal parameters will be synthesized with the aim to be employed in water desalination process using simple adsorption technique. Due to numerous variables (i.e. pH, treated water temperature, concentration of ions, mass and size of zeolites etc.) that can affect the sorption process during water treatment, it will also be taken into consideration during the analysis.

2. Environment Issue and Access to Clean Water

Environmental protection has become a national issue as well as international issue in many years. Environment issues become more complex and challenging due to water pollution, solid waste management, soil contamination, hazardous waste management, and many more. According to Olaniran [21], water pollution can be described as excessive amount of impurities found in water bodies that are dangerous for usage or consumed by any living things. Water pollution has become the primary reason of clean water sources scarcity. To simplify, pollution was mainly caused by human activities, especially due to direct discharge of industrial and commercial waste into river streams and also due to modernization and globalization.

Based on Muyibi *et al.*, [22], the pollution world record has shown that there is an increment of water pollution around the world due to various industries activity. This serious pollution problem has led to the increment of water treatment processes that needs to eliminate various toxic chemical and dangerous substances. Previously, reverse osmosis has been used intensively to treat the seawater [23]. However, reverse osmosis took longer time to filter the water. This RO system was taking about 3 to 4 hours to filter just one gallon of water. In addition, RO is an expensive system, including the installation process. Therefore, the solution to gaining fresh water from the seawater would be the best approach since seawater contains less contaminate and abundantly available. Cao et al. [24] in their work suggested that water desalination using membrane technique is energy-inefficient and expensive, and some use membranes that operated at a high temperature. Therefore, the usage of simple and cheap adsorption techniques is expected to reduce the cost and offer a simple and easy operating system as compared to other treatment techniques. For other water treatment process such as distillation process, it requires a large amount of energy and water.

2.1 Water Treatment

Water treatment can be defined as set of processes done in order to purify water sources and improves water quality to appropriate level and safe for daily use. Water treatment usually done in water recreation, maintenance of river stream flow, industrial water supply and many other uses. It helps to remove pollutants and unwanted components from water and lowered the impurities concentration, depending on its desired end-use. In addition, water treatment for drinking water is to make sure that dangerous contaminants have been completely remove from the raw source so that no adverse health effects, whether short term or long-term risk would happen to consumers. Examples of pollutants particles that can be found during drinking water treatment are mainly minerals (e.g. iron, manganese and etc.), suspended solids, algae, viruses, bacteria and also fungi. The removal of these contaminants involves both physical and chemical processes. Physical process includes settling and filtration whilst chemical process includes disinfection and coagulation. Techniques employed in treatment of water has its own pros and cons in term of cost and energy. Because our water stream is highly contaminated, gaining fresh water from seawater is the best alternative. Technique of gaining clean water from seawater is known as desalination.

2.2 Desalination

Desalination technology is mainly used to eliminate salts from sea water to obtain fresh water supply. Desalination has been introduced since aeon, whereby the Greek sailors removed salts from sea water via boiling and evaporation process. As for Romans, the trapped salt using clay-based filter. Current desalination system still applied the concepts used by the Greek and Romans. To date, there are approximately 15000 desalination plants that were located globally with the biggest plant to be found in Middle East countries. Furthermore, with the increasing needs of high quality and clean water sources, low-cost approaches combining desalination and ion exchange would be advantageous. Desalination method falls into three categories; (1) chemical, (2) thermal (evaporative) and (3) membrane-based methods. Processes used in desalination include vapor compression, multistage flash (MSF), electrodialysis, reverse osmosis, solar distillation, freezing, ion exchange, solvent process, and others. For MSF process, it was well known since decades ago in which Weirs of Cathcart pioneered the technology at early days. MSF process promoted lower operating cost and has the ability to be operated on low grade steam, making significant development in 1960s following its beneficial characteristics [25]. Further proof can be seen globally where 64% of total desalinated water from all over the world was produced by MSF method with plant located in Middle East countries as the majority that employed the concept [26]. Similar with membrane technology, MSF is also considered as energy-inefficient because it uses thermal energy and mechanical energy [26]. Other process known as vapor compression process has also been widely investigated to enhance the efficiency by addition of unit capacities and reduce consumption of energy [27]. Still, adsorption beats all the other methods and have been successfully utilized in separation industries with numerous types of adsorbents being introduced since then. It is energy-efficient, simple to operate, economical and is very reliable.

2.3 Sea Water

Ocean water contains dissolved salt that gives different properties than pure water. It was discovered that sea water is denser than fresh water and pure water due to its salinity that added to the total mass rather than to the volume. Content of salt in sea water is approximately 3.5% (35 g/L). As the salt became concentrated, freezing point of sea water decrease. Typically, the freezing point is at ~2°C while the surface density is at range 1020-1029 kg/m^3. However, down the ocean at increasing pressure, the density of water can reach over 1050 kg/m^3. For pH, it usually is limited to 7.5 to 8.4. Some ions found in sea water are H_2O, Cl^-, Na^+, Mg^{2+}, Ca^{2+}, SO_4^{2-}, and others. Castro and Huber [28] addressed composition of sea water as in Table 1 below.

Table 1
Seawater composition [28]

Element	Concentration in seawater (ppt)	Percentage composition (%)
Cl	19.35	55.03
Na	10.75	30.59
SO_4	2.70	7.68
Mg	1.30	3.68
Ca	0.42	1.18
K	0.40	1.11
HCO_3^-	0.15	0.41
Br^-	0.07	0.19
BO_3^{-3}	0.03	0.08
Sr^{+2}	0.01	0.04
Fl^-	0.001	0.003

2.3.1 Adsorbent material: Zeolites

Two adsorbent materials that are abundantly available on earth are soil and clay minerals. Al-Jlil and Alharbi [29] stated that adsorption test on Saudi Bentonite clay and RO method has shown efficiency result percentage of 88.89 and 87.92 respectively. In terms of economy, RO-unit operations were far expensive compared to Saudi bentonite clay. Furthermore, the clay does not need to undergo regeneration for the next adsorption cycle while frequent cleaning needs to be done to membrane modules as fouling effect takes toll on the adsorption efficiency of the modules. This led to addition in total cost of operation. In addition, Al-Kharabsheh [30] reported that production of 5 gallons of treated water would waste 40-90 more water gallons. The capability of soil and clay mineral to be functioned as adsorbent candidate which does not have too much different with commercial materials and water treatment technology have open widely the development and modification of clay. This including the modification of clay to zeolite. Therefore, the use of zeolite as adsorbent synthesized from natural zeolite will provide a sustainable and greener approach. Usage of zeolites in desalination process has been investigated by various scientists with a reported complete reduction of pollutants (99.99%) in series of columns filled with zeolites [11]. However, it still depends on zeolite type and salinity of water [31]. Since seawater is high in sodium ions, the use of a Na-free zeolite will be recommended for the removal of sodium ions. It can be said that zeolite-based membranes for desalination are one of novel application in elimination of contaminants from sea water even though there are existing inorganic membranes that were applied to concentrated radioactive solutions through RO and membrane distillation [32,33].

2.4 Adsorption technique

Adsorption is considered as the most promising approach for water remediation and desalination due to its energy-efficient, economical and simple operating conditions in reducing or removing contaminants from effluents. Surface solid in which the pollutants would bind onto is known as adsorbent. Both organic and inorganic suspensions can be removed from raw water source through adsorption. Adsorption is an exothermic process, meaning that energy would be liberated during the process. According to Bushra and Shahadat [34], adsorption system involves the sorption process of contaminants' molecules on adsorbent surface through interactions between molecules and diffusivity of adsorbate molecules. Key parameters that influence adsorption efficiency are adsorbent dosage, initial concentration, pH, temperature, size of particles and surface morphology. It also depends on the sorbate structure. Figure 1 illustrates the adsorption process. Adsorption can be breakdown into two; physisorption and chemisorption. Physisorption, or known as physical adsorption occurred when forces presence between adsorbate and adsorbent are weak Van Der Waals forces. The adsorbate (gas or liquid) molecules which attract onto the adsorbent (solid) surfaces are primarily held at the micropores in their majority and some extent to mesopores of the solid adsorbent. These phenomena are physical in nature, and hence, this process can be reversed using heat or pressure, etc. For chemical adsorption or chemisorption, attraction force is given by chemical reactions between adsorbate and adsorbent resulting in chemical bond formation not completely reversible as physical adsorption [35]. Since physical adsorption is due to attraction forces, heat is released due to the change in the energy level of the 23 adsorbate molecules between gaseous and adsorbed phases; therefore, physical adsorption is an exothermic process [36].

Fig. 1. Adsorption process

2.4.1 Mathematical model

Important factors in optimizing adsorption system to remove contaminants from water is by understanding the relationship between the adsorbed pollutant per unit weight of sorbent (q_e) and remaining concentration of pollutants in testing solution at equilibrium (C_e). Many isotherm models were developed previously such as Freundlich, Langmuir, Halsey, BET, intraparticle diffusion and Redlich-Peterson in order to evaluate the results and performance of the experimental adsorption. Langmuir isotherm model is utilized when adsorption happened on specific homogenous sites present in the adsorbent surface, usually in monolayer adsorption [37]. Equation for Langmuir model at equilibrium is:

$$\frac{C_e}{q_e} = \left(\frac{1}{b}\right)\left(\frac{1}{K_L}\right) + \left(\frac{1}{b}\right)(C_e) \quad (1)$$

where C_e is concentration of remained solution (mg/L), q_e is adsorbed pollutant per unit weight of sorbent (mg/g), b is the capacity of monolayer and equilibrium constant for Langmuir model is denoted as K_L. Plotted graph for $\frac{C_e}{q_e}$ versus C_e should produce a straight line with gradient of $\frac{1}{b}$ and point interception at $\left(\frac{1}{b}\right)(K_L)$. On the other hand, Freundlich model isotherms is commonly used for multilayer adsorption over a heterogenous surface [37]. The model isotherm equation is as follow,

$$q_e = (K_F)(C_e)\left(\frac{1}{n}\right) \quad (2)$$

From Eqn 2 above, K_F is the constant for Freundlich model while $\frac{1}{n}$ is the intensity of sorption or known as Freundlich coefficient. If the value of n is equal to 1, it should be noted that partition between two phases is in independent, regardless the value of its concentration.
To find out the rate of adsorption and its mechanism of adsorption, calculation via two kinetic models that are known as Pseudo First Order and Pseudo Second Order were applied [38]. Kinetic models are introduced for adsorption rate analysis of adsorbate to the sorbent surface. For Pseudo First order, Lagergren's equation in equation 3 below was used [39]:

$$\ln \frac{q_e}{q_t} = \ln q_e - K_1 t \quad (3)$$

where q_e and q_t are the adsorbed amounts at equilibrium and at time=t (mg/g), respectively. K_1 is the constant for first order reaction (1/min). Values for K_1 and q_e were determined from graph of ln (q_e-q_t) versus t conducted at various concentrations. K_1 values are from the slope while q_e are from the interception points. Next, for Pseudo Second Order, assumption is the surface adsorption which involves chemisorption as the rate controlling step, and pollutant elimination from aqueous solution is caused by the physiochemical interactions between the phases [38]. Equation for Second Order kinetics:

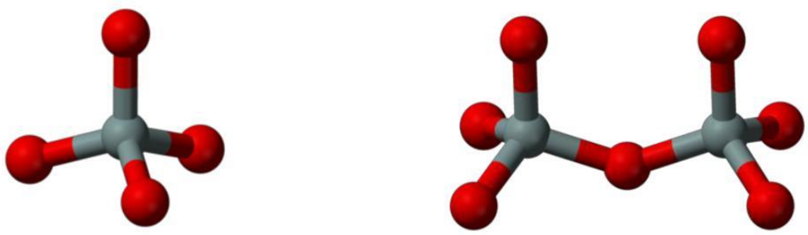
$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e}(t) \quad (4)$$

$$h = K_2\left(q_e^{\,2}\right) \quad (5)$$

h is the initial adsorption rate (mg/g.min) and K_2 is the constant for pseudo second order reaction rate (g/mg.min).

2.5 Zeolite

Zeolite is an inorganic crystalline material with three-dimensional framework structure consists of aluminosilicates, with comprising cations and water molecules in its framework [40]. Cations present in the zeolite can be changed by other cations present in the solutions while the intra-crystalline zeolitic water can be eliminated reversibly [40]. The ability of zeolites formation into various type and structure that can be used for various application strongly depends on the different composition of raw materials. Differences in zeolites formation are mainly controlled by the Si/Al ratio. In 1984, Riberio *et al*. [41] has addressed the preparation of zeolites for application in water remediation in their published literature. According to Sean and Yoshio [42], zeolites are group of hydrated aluminosilicates of the alkaline earth metals such as Na, K, Mg, Ca, Li and Ba. It refers to crystal aluminosilicate with a corner sharing TO_4 tetrahedra (T=Silicon or aluminium) producing a 3-Dimensional and four-connected framework consist of uniform pores sizes of molecular dimensions. Figure 2(a) demonstrated the primary unit of zeolite structure that has a central atom either silicon or aluminium and four oxygen atoms surrounded the central atom. When oxygen atom is shared by two tetrahedra, tetrahedron structure will be obtained as in Figure 2(b).



(a) (b)

Fig. 2. (a) TO_4 tetrahedra; (b) Tetrahedron [43].

In 1896, Friedel [44] suggested that dehydrated zeolites comprised of open spongy frameworks structure. It can be identified by five characteristics as listed below [45,46]:

1. Catalytic behaviour
2. High hydration propensity
3. Stable crystalline structure
4. Low density and high volume of void
5. Exchange of cation and sorption behaviours.

Porous zeolite particles are grouped by its pore sizes which are micropores and mesopores. Microporous particle have diameter of less than 2 nm and zeolites are crucial member of this group type [47]. Figure 3 illustrated the zeolites having a sponge-like image due to its regular structure and porous size. The presence of pores helps to hold water molecules and other molecules.

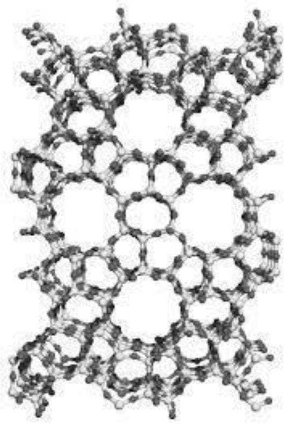


Fig. 3. Structure of Zeolite silicalite showing its pores [43].

2.5.1 Application of zeolite

Usage of zeolite has been increasingly widespread especially in industries as it can be applied in various applications. Current industries utilized the zeolites for gas purification and separation, catalysis reaction, wastewater treatment, radioactive waste treatment that includes water desalination and also ion exchange.

2.5.1.1 Ion exchange

Ion exchange is the removal of ions from water or other solutions and replaced by other ions. According to Alchin [48], ion exchange does not change the physical structure of the material. This approach can efficiently introduced protons and ions into zeolites at ambient environment with a very low impact to the zeolites structure [49]. Zeolites are not soluble in water and holds an opposite charge at the exchange sites. Zeolites can also possess ionic sieve properties due to narrow diameter channel that does not allow certain cations to pass or the cations exceed certain size that cavity dimensions are inadequate to accommodate the number of available exchange sites. One important characteristic of zeolites is its capability cation exchange. Zeolites are advantageous, especially as low-cost options, and the method can be cost-effective. Top [50] stated that exchange behaviour of cations in zeolites are dependent on its cationic nature, size, charges of hydrated and anhydrous species, temperature, cation concentration, structure of the zeolites itself and many more. Unwanted ions in aqueous solution are taken out and replace with appropriate ions. A German scientist in 1905 first employed synthetic aluminosilicate as water softeners. It denoted the first softeners that use the concept of ion exchange. Even though aluminosilicates are hardly used nowadays, the term “zeolite softener” is still used to define cation exchange mechanism. Environmentally friendly methods and materials for desalination are available via ion exchange. Desalination can effectively eliminate anions and cations in separation steps with minimized generation of caustic waste. Similarly, zeolite can be applied in desalination due to their specific ion exchange behaviour. It has the ability to be operate at specific temperature and pH desired for economical and energy-efficient process. Zeolites are an enhanced ion exchange material that have high ion binding capacity in particular for monovalent ions and favourable regeneration properties.

2.5.1.2 Adsorption

Numerous approaches have been developed for removal of toxic contaminants. Popular method include adsorption, ion exchanger, chemical precipitation, reverse osmosis and etc [51]. Zeolites were chosen as ion exchange materials due to its superior structural properties and other valuable characteristics. Natural zeolites are abundant and known to possess a high selectivity for heavy metal contaminants [52]. Previously, researchers conducted a study to investigate the performance of Brazilian natural zeolites for cation-exchange with Cd^{2+}, Ni^{2+}, Cr^{3+} and Mn^{2+} in synthetic wastewater [53]. They found out that the material has a good heavy metal ions removal performance. Chunfeng et al. [54] synthesised pure Cu-Zn gel zeolites and compared the resultant heavy metal removal performance with commercially available zeolites. Next, two natural zeolites; mordenite and clinoptilolite, and a modified zeolite were used to eliminate As(V) contained in water [55]. Results revealed that modified zeolites showed a better performance in As(V) removal than the natural zeolites. To recover Europium(III), Iron(III) and Thorium(IV) from solutions, Sharma et al. [56] synthesised a heulandite zeolites. From the results, it was known that Eu(III) and Fe(III) were more favoured by the zeolites. Industrial establishments are obliged to treat the heavy metal content specified by law and reduce their concentration to acceptable level. Emami Moghaddam et al. [57] stated that zeolite-immobilized chlorella can be used in various applications include remediation of wastewater and production of biofuel. Great numbers of researcher have been used various techniques for removing pollutants especially heavy metals from water/wastewater because of scarcity in water resources and increasing pollution levels. As adsorption technique is simple and can remove diverse pollutants, adsorption is preferable. The present study suggests that various types of adsorbents, including natural materials such as bio absorbents, clay minerals, and some of the materials modified to improve their adsorption capacity including carbon-materials, metal oxides are summarized. The metal ions adsorption amounts onto various adsorbents and under different experimental conditions besides the relationship between adsorption properties and capacity was discussed. When considering the adsorbents economy, recyclable, effective and low-cost, adsorbents should be synthesized for its extensive application in our daily life. In the future, eco-friendly adsorbent materials that have high sorption ability and high surface area, easy separation from aqueous solution and comprised of suitable functional groups, they may be preferred. Adsorption process and materials that are cheap, practical, and efficient should be explored further for the possibility of heavy metals recovery and desalination of sea water.

2.5.1 Hydrothermal method: Zeolite synthesis

Increasingly popular method to synthesize zeolites is by hydrothermal method which garnered attentions from scientists and technologist from different disciples. Hydrothermal method is a heterogenous reaction in a solution that was conducted above room temperature and pressure higher than 1 atm [58]. As for Rabenau [59], hydrothermal synthesis is experiment that were carried out in aqueous media at temperature more that 100°C and 1 bar. Hydrothermal method is included in the most efficient techniques in synthesizing finer particles at stable conditions with a simple approach and low operating cost. This technique was used from long ago until now to get various kind of nano-sized and finer particles that have functional and superficial properties which contribute to many crucial applications. In this present work, the synthesize of multifunctional of zeolite materials will be conducted at a controlled processing parameter. Zeolites can be prepared from various sources such as fly ash, kaolinite, and rice husk. More than 150 zeolites have been synthesized by experimental work and approximately 40 natural zeolites were known, obtained from volcanic ash

[45]. Basically, man-made zeolites are manufactured from silica, aluminium oxide and water under high temperature [60]. First laboratory synthesized zeolite known as levyne (hydrated silicate mineral) was produced in 1862 by Deville, where potassium silicate and sodium aluminate were heated in a glass ampoule [60,61,62].

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