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Review

Recent Findings on Fly Ash-Derived Zeolites Synthesis and Utilization According to the Circular Economy Concept

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Abstract: The synthesis and utilization of zeolites derived from fly ash (FA) gained significant attention years ago due to their potential to address environmental challenges and promote sustainable practices subscribing to the circular economy concept. This paper highlights the recent findings regarding the synthesis and utilization of zeolites derived from FA. It begins with a discussion about the recent challenges regarding industrial waste management and statistics regarding its availability on the global market with a special insight into the situation in Poland. The characteristics of FA obtained from various fuels were presented and the main differences were highlighted. Then, different methods used for the synthesis of zeolites from FA were discussed in small and pilot scales taking into consideration the main challenges and problems. The analytical methods used in porous materials synthesis verification and properties determination were described. The sorption properties of FA-derived zeolites were presented and discussed. Finally, the paper emphasizes the potential applications of fly ash-derived zeolites in different fields. Their importance as sustainable alternatives to conventional materials in industry, construction, agriculture, power, medicine, and other industrial sectors was analyzed.

Keywords: fly ash; waste management; zeolite; circular economy

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1. Introduction

The environment has been negatively affected by the exponential growth of our economy as well as our heavy dependence on conventional energy sources. The degradation of the natural environment leads to severe climate changes. Another significant global concern is the proper management of waste, especially industrial ones, like fly ash from coal [1] and biomass-firing power plants [2,3].

Fly ash (FA) is a solid residue of fuel combustion in power boilers. It has a complex composition depending upon the raw feedstock properties [4]. FA is mostly composed of aluminosilicates [5] forming fine particulate matter captured using dust removal equipment, ranging in size from 0.5 μm to 300 μm [6]. FA might be used directly as a waste material or after preprocessing, like for example in zeolite synthesis. Zeolites are well-defined crystalline aluminosilicate porous materials, built up with a framework of alumina and silica tetrahedrals [7]. These porous materials are composed not only of aluminum and silica, but also oxygen, water, and cations such as Na^+, K^+, Mg^{2+}, and Ca^{2+} as counterions [8,9]. Their general chemical formula is $M_{y/n}[(AlO_2)_y(SiO_2)_m]_zH_2O$ [10], but water within their structure can be removed with the application of heat [11]. In the tetrahedral structure, zeolite includes silica (Si^{4+}) and aluminum (Al^{3+}) cations, which creates large negatively charged channels. Therefore, the mechanism of adsorption using zeolites was identified to be ion-exchange [12]. Additionally, their unique micro- and mesoporous structure, high ion-exchange capacity, substantial specific surface area, and thermal stability contribute to their prominent position in the industrial market [7].

Zeolites can be obtained from natural sources or synthesized using various methods [9]. There is a diverse array of natural zeolites, with more than 40 different varieties known.

Among these, the most frequently encountered types are analcime, chabazite, heulandite, phillipsite, and stilbite [13].

The evaluation of synthetic zeolite production using various materials like clay minerals, low-carbon materials, and FA has created new value-added products that find applications in various branches of industry, agriculture, biochemical, and chemical sectors such as catalysis [14,15], water treatment [16,17], and gas storage [18,19]. Utilizing FA as a feedstock for zeolite synthesis aligns with waste minimization strategies and the circular economy concept, but it requires specific procedures [20–22]. Commonly used and well-known synthesis methods include hydrothermal synthesis [23], the molten salt method, alkali activation, and microwave synthesis [24,25].

This study focuses on analyzing FA-derived zeolites for their potential use as adsorption materials in various industries. Recent findings in zeolite synthesis and utilization are analyzed and presented.

2. Industrial Waste Management in Poland

2.1. Industrial Waste Generation Globally and in Poland—FA Generation and Utilization

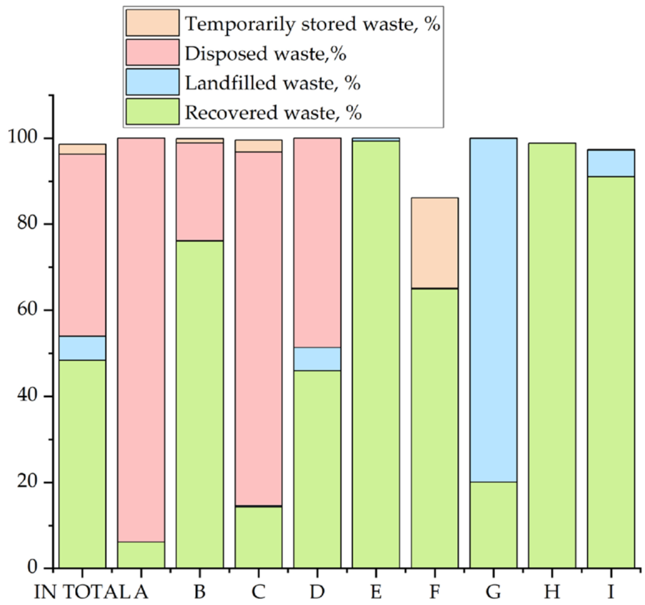
The largest global coal fly ash (CFA) producers are India, China, USA, Russia, and the EU [4]. China's annual production of FA is equal to almost 500 million tons, but only about 40% of this quantity is effectively utilized [26]. In India more than 230 million tons of FA is generated but the waste is properly managed and 92% of FA is utilized [27]. In the USA, FA generation has been decreasing since 2014, whereas the amount used in other sectors is increasing and in 2021 more than 60% of coal combustion products were further utilized [28]. A very low CFA utilization rate is observed in Russia, only 8% of CFA is used [29].

Poland is one of the countries with the relatively lowest production of municipal solid waste in the European Union [30]. Unfortunately, due to our heavy dependence on fossil fuels in the production and energy sectors, there is a noticeable generation of industrial waste. According to the Environment 2022 report published by Statistics Poland in 2021, a total of 107.7 million tons of waste (excluding municipal waste) was produced, with over 61.9% coming from the mining and extractive industries. As much as 48% of the waste was recovered, but only 7% was disposed using methods other than landfilling, and a significant 44% of waste was landfilled (Figure 1). It is essential to remember that there are also over 1811.4 million tons of waste currently being stored in Poland [31]. However, only 10% of the total amount of FA generated each year is landfilled in Poland. Whereas in China only 30% of CFA is used, mostly in building materials and the large amount of 3 billion tons of CFA is landfilled [32].

In Poland, the largest amounts of industrial waste are produced by industrial processing plants (46.9%), as well as the mining (31.8%) and energy (18.6%) industries. Consequently, the regions with prominent industrial activities generate the most significant quantity of waste, mainly from metallurgical, mining, and energy sectors. The storage of industrial waste leads to health hazards due to potential exposure to toxic and harmful substances present in the waste. Industrial waste often contains heavy metals, as well as aliphatic and aromatic hydrocarbons, and pesticides, all of which have physicochemical and toxic properties that can endanger human health and the environment [33].

The improper location, construction, and operation of landfills, along with uncontrolled storage of industrial waste, create opportunities for the release of chemical substances from the accumulated waste, leading to potential threats to the environment [33].

The efficiency of the recovery process depends on the waste's properties, its consistent composition, and the level of contaminants present. In the case of waste generated during the rinsing and purification of minerals, slag from smelting processes, soil and stones waste, as well as mixtures of FA and solid waste from wet flue gas desulfurization and FA from coal combustion, the recovery process is quite effective. However, for waste resulting from the flotation enrichment of non-ferrous metal ores and mixtures of ash-slag from the wet disposal of combustion residues, the recovery process is conducted for less than 20% of the generated waste (Figure 1).



A	Waste generated during the rinsing and purification of minerals
B	Mixtures of ash and slag from wet disposal of combustion residues
C	Waste from the extraction of non-metallic minerals other than metal ores
D	Slag from smelting processes
E	Soil and earth, including stones
F	Soil and earth, including stones
G	Clarified water sludges
H	Mixtures of fly ashes and solid waste from calcium-based flue gas desulfurization methods
I	Coal fly ashes

Figure 1. Industrial waste generated and landfilled in Poland, data for 2021 [31].

The high utilization of FA in the industry is due to changes made in Poland since 1965; new regulations introduced the possibility of FA-doping cement [34]. This addition improves the properties of concrete and increases the utilization of this waste in the industry [35]. However, the use of fly ash as a substitute for cement is limited by the amount of free lime in the ash [36]. The chemical and physical properties of silica FA used as an additive in concrete production have to be standardized [4]. It was observed that the addition of FA and zeolite to the cement slightly decreases the compressive strength of the material. However, it was also noted that incorporation of FA and zeolites improves the flowability of concrete [37].

FA fulfilling the requirements described in the standard serves as a valuable additive in the production of commodity concretes, precast concrete products, dry mixes, and mineral adhesives [38]. CFA can also be used in the production of concrete-ash mixtures used in road construction. However, due to the increasing co-firing of conventional fuels with other fuels (such as biomass or alternative fuels) in power plants, as well as the use of flue gas treatment systems, such as sorbents dosed directly into the combustion chamber or flue gas ducts, which directly affect the composition of FA, new opportunities for using this waste in the industry are constantly growing. FA can be directly used in agriculture as fertilizer [39], or if it contains large amounts of carbon and phosphorus, FA can be used as a modified material in the production of mineral-organic fertilizers [5,19,40] or as material for the synthesis of potassium zeolites [41]. Methods for synthesizing advanced porous materials from FA are also being developed, such as commonly used zeolites [19,42,43] and metal-organic frameworks (MOFs) [44]. Suitable treatment also allows for the use of FA from municipal waste incineration in phosphorus recovery processes [45].

FA which is rich in carbon is also a valuable adsorbent for capturing heavy metals, such as mercury, from flue gas streams [46], or heavy metals like copper and nickel in water treatment systems [47]. The disposal of wastewater containing dyes into aquatic streams poses a significant challenge due to its adverse impact on the water ecosystem. Direct dyes with their complex aromatic structure make their separation from industrial wastewater a difficult task. However, a promising solution was found with the use of chemically modified CFA, which demonstrated a noticeable removal efficiency for these types of contaminants [48]. Additionally, after hydrothermal treatment, FA can be used as a hydrophilic material with a high potential for use in solar air-conditioning systems [49].

In recent years, there has been a growing trend of using ashes from biomass combustion to enhance soil quality, both in agricultural practices and in remediation and reclamation techniques. There is also consideration for using ash as a binding additive in the production of fertilizers from other waste materials, such as sewage sludge [50].

The method of treating FA for its utilization in industry is largely dependent on the combusted fuel or fuel mixtures, combustion technology, and applied flue gas cleaning methods [51]. Co-firing biomass and high unburned carbon content create possibilities for using FA in agriculture. However, a significant barrier is the contamination of FA with heavy metals when co-firing fossil fuels with alternative fuels. In such cases, the FA needs to be classified as hazardous waste if it contains high levels of heavy metals. Monitoring the stability of specific contaminants is also necessary due to the potential for uncontrolled environmental pollution.

2.2. FA Types and Properties

During the combustion process of fuels in power boilers, two types of ashes are obtained: bottom ash (constituting 10 to 20% of the total ash mass) and FA (70 to 90%). The proportion of each type depends primarily on the combustion technology and the type of fuel being burned. Besides ashes, other solid by-products of combustion include desulfurization products, slag, gypsum, and other residues from flue gas cleaning processes [52].

The standard ash analysis generally determines the mineral composition of the solid residue remaining after the combustion process. Typically, elements such as silica, aluminum, iron, calcium, magnesium, sodium, potassium, manganese, phosphorus, and titanium are considered, along with any specific trace elements in certain cases [53–55]. For example, in ashes from the incineration or co-incineration of waste, the content of heavy metals is of key importance [56]. The main components of the mineral phase are expressed as the highest oxides of these elements, assuming that all bonds and compounds present in the raw fuel sample are destroyed during the combustion process, converting into oxide form. Table 1 presents major FA constituents, mainly oxides depending on the combusted fuel. The classification of FA is based on the sum of selected elements in oxide form, leading to three main groups:

1. Silica-based, determined as a sum of the concentrations of selected oxides: SiO_2 + Al_2O_3 + TiO_2
2. Calcium-based, determined as a sum of the concentrations of selected oxides: CaO + MgO + Na_2O + K_2O
3. Iron-based, determined as a sum of the concentrations of selected oxides: Fe_2O_3 + MnO + SO_3 + P_2O_5,

Intermediate groups are also distinguished based on the concentration of individual elements in the mineral phase [57].

Table 1. Major FA constituents depending on the combusted fuel.

FA Component	PL-L1	PL-HC 2	PL-L3	PL-B4	PL-B5	PL-W6	PL-W7
SiO_2	27.37	48.43	47.3	36.03	44.41	14.02	5.55
Al_2O_3	6.63	28.72	31.4	8.33	10.80	22.73	18.19
CaO	34.48	4.63	1.7	27.41	23.84	20.22	32.49
MgO	8.23	2.60	1.9	3.56	3.76	6.53	2.29
Na_2O	1.08	1.77	-	0.87	1.27	5.83	5.55
K_2O	0.41	2.81	-	4.92	3.99	3.08	3.22
Fe_2O_3	3.75	6.35	7.7	4.12	3.63	0.56	0.62
TiO_2	0.96	1.17	1.6	0.94	1.05	0.76	0.76
P_2O_5	-	-	-	3.21	2.02	0.82	1.23
Heavy metals (sum)	-	-	-	-	-	10.49	7.48
Reference	[58]	[59]	[60]	[3]	[61]	[62]	[62]

PL-L1—FA obtained from lignite combusted in a pulverized coal boiler (Poland).

PL-HC2—FA obtained from hard coal combusted in a pulverized coal boiler (Poland).

PL-L3—FA obtained from lignite combusted in a fluidized bed boiler (Poland).

PL-B4—FA obtained from a pulp and paper mill, from a boiler fueled with waste forest biomass (Portugal).

PL-B5—FA obtained from biomass combusted in a fluidized bed boiler (China).

PL-W6—FA obtained from a grate boiler in a waste incineration plant (Italy).

PL-W7—FA obtained from waste combusted in a fluidized bed boiler (Italy).

Major constituents of FA are usually silica and aluminum, but in some cases calcium is also one of the main components of the mineral matter, especially in the case of biomass and waste. Additionally, in the case of biomass a noticeable content of alkali metals, mostly potassium, might be noted. FA collected from the waste incineration process is usually contaminated with heavy metals which might affect its potential use in industry.

3. Circular Economy (CE) Concept

In 2015, a circular economy (CE) package was published by the European Commission to enhance the transition from a linear to a circular economy, where resource efficiency is increased and the value of products and materials is maintained as long and as productively possible (Figure 2) [63]. In the CE concept, when a product cannot be further used, options like remanufacturing and recycling are promoted to create additional value. The economic benefits associated with the CE concept are connected to additional job creation and the promotion of innovation and environmental benefits [64].

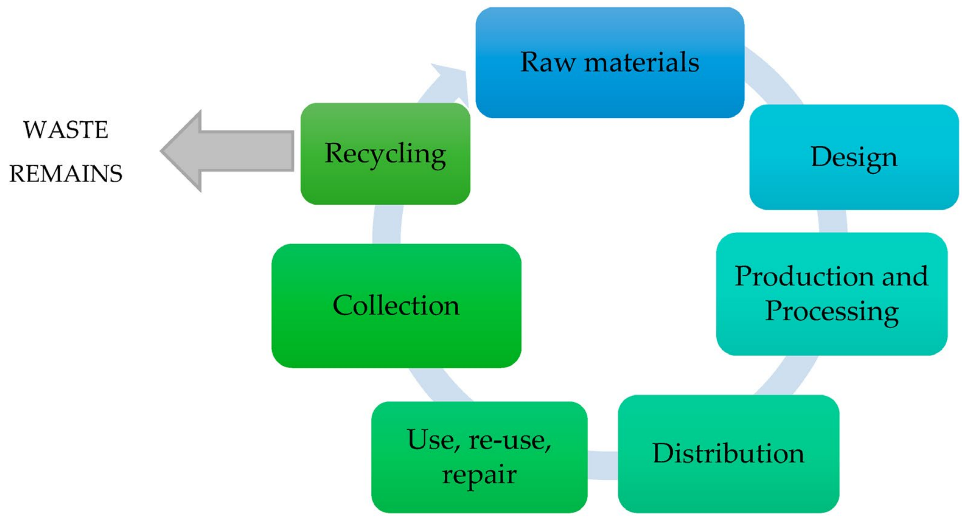


Figure 2. General idea of the circular economy concept.

Society has been aware for the last several years that the increasing consumption of non-renewable material resources cannot sustain human development anymore; resources must be utilized at their maximum. Until now, many intensive environmental problems have arisen, and some boundaries that defined the safe environment for humans have already been transgressed. Very soon, we will be challenged by the running out of global fossil fuel resources. However, pollution and climate change are even more influential factors forcing our society to change its habits.

The awareness of this situation led to changes in waste processing from a linear approach to a circular, which is depicted by sequential circles. Earlier, linear waste processing was based on the traditional take–make–consume–dispose approach (Figure 3).

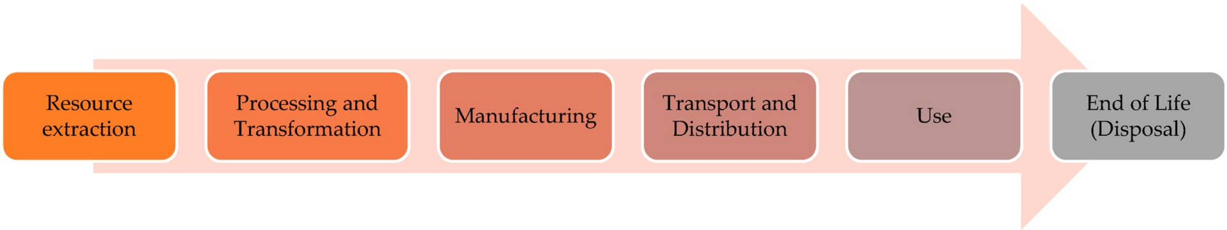


Figure 3. General idea of the linear economy.

The CE is an economic model that aims to maximize resource efficiency and minimize waste by keeping products, materials, and resources in circulation for as long as possible. The concept of the circular economy is based on the idea of closing the loop of production and consumption, where products are designed to be durable, repairable, and recyclable, and where waste and emissions are reduced to a minimum [65–67].

The CE concept has gained attraction globally as a promising solution to address environmental challenges and create a more sustainable and resilient economic system. It encourages businesses, governments, and consumers to adopt more circular practices, which can lead to reduced resource depletion, less waste generation, and a more inclusive and sustainable economy.

CE strategies are widely described in the literature, but the official definitions of the strategies promoting a CE are not defined. The most developed R-framework uses ten strategies to increase circularity: refuse, rethink, reduce, reuse, repair, refurbish, remanufacture, repurpose, recycle, and recover. CE strategies were designed to preserve raw materials and decrease their consumption [68].

The seven pillars of the CE supporting the evolution of economic rules and incentive structures that actually fulfill the end results were proposed by Metabolic [69]. The most efficient and successful technologies and business models support all seven pillars at the same time, but not all of them are equally important. Some of the most important aspects of the pillars are:

- long-term consequences and irreversibility,
- making the ability of our planet to provide a safe space for us weaker,
- high uncertainty of outcomes.

The seven pillars of the CE help us approach problems in a systemic manner. They might be translated into quantitative tools: metrics and indicators. These are useful tools for the evaluation of the circularity of products, projects, businesses, etc.

The CE concept is already implemented in some big companies like Canon, Renault, Philips, or Tata Motors Limited, which are collecting used equipment to remanufacture, recycle, or reuse it and resell with the same guarantee as brand new products [70]. Such actions lead to the minimization of waste generation and the preservation of natural resources. The circularity is also enhanced by extended guarantee of the products and encouraging customers to repair broken and used goods instead of buying new ones.

4. FA-Derived Zeolites

4.1. FA as a Potential Precursor for Zeolites Synthesis

Zeolites are a group of aluminosilicate minerals containing oxides of alkali metals and alkaline earth metals in their composition. They belong to the class of so-called molecular sieves. Depending on their origin, they can be classified as natural or synthetic. Natural zeolites are formed as a result of the weathering of volcanic rocks. Synthetic zeolites, on the other hand, form a much larger group of materials, with over 150 types. Many synthetic zeolites have a similar structure and geometric arrangement as natural zeolites [5]. However, synthetic zeolites have better properties and a lower contamination content than natural ones [71].

The most commonly used zeolites in industry are of type A, X, and Y. Figure 4 shows the morphology of commercial molecular sieves 4A and 13X. Type A zeolites are characterized by relatively low silica content, with a molar Si to Al ratio below 2. Type X and Y zeolites, unlike type A zeolites, exhibit increased resistance to acids and high temperatures. For type X zeolites, the silicon module does not exceed 2, while for type Y zeolites, it ranges from 2 to 5 [72]. The artificial synthesis of these materials has improved their heat conduction properties. Satisfactory results have been obtained in the synthesis of zeolites from natural aluminosilicates like halloysite [73,74] and kaolin [75], synthetic materials like kaolinite [76], as well as combustion by-products like FA [77–79]. To achieve specific properties, zeolite materials undergo additional modifications. Table 2 presents the properties of reference materials—selected natural and commercial zeolites described in the literature.

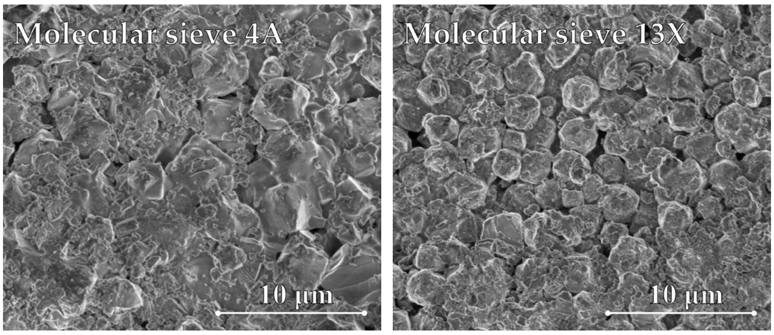


Figure 4. Scanning electron microscope images of molecular sieves 4A and 13X.

Table 2. Comparison of specific surface area and pore diameter values for natural and commercial zeolite materials.

Adsorbent	Specific Surface Area, m^2/g	Pore Size, nm	Reference
Natural zeolite	9–15	9.2–14.6	[80,81]
Zeolite 4A	7–559	22	[82–85]
Zeolite 13X	310–730	1–2.5	[84,86–88]
ZSM-5	304–417	-	[89–92]
TS-1/2zeolite	500	-	[93]
Zeolite Y	527	2.5–3	[92,94]
Zeolite HY	620–625	7.4	[95,96]

Natural zeolites are characterized by very low specific surface area resulting in weak and moderate sorption capacities. However, synthetic zeolites which are very frequently used in the industry might be characterized by a specific surface area several times higher, mostly due to microporous structure development.

4.2. Methods of Synthesis for FA-Derived Zeolites

One of the most innovative applications of FA is the synthesis of sorption materials such as zeolites. FA-derived porous materials might increase the amount of this industrial waste management according to the CE concept. FA has a similar chemical composition to zeolites, although the combustion and co-combustion of biomass and waste fuels affect the composition of the mineral phase, which may affect the properties of the obtained porous materials. The production of new porous materials from heterogeneous materials with variable composition, such as FA, forced the adaptation of existing and the development of new special procedures dedicated to the synthesis of zeolites from FA. There are many synthetic methods, which vary depending on the temperature and time of the process (hours/days), environment, Si to Al ratio, reaction components ratio, process steps, etc. The most commonly used and well-known methods for the synthesis of zeolite materials are hydrothermal synthesis, the molten salt method, alkaline activation, microwave synthesis [97,98], and ultrasonic treatment [99]. Hydrothermal crystallization consists of three stages of synthesis: dissolution, condensation, and crystallization in an alkaline solution with a pH > 8.5. Typically, the process is carried out in autoclaves with increased pressure [100]. Zeolite samples prepared via the hydrothermal method using CFA as a pre-cursor were analyzed in [101]. It was noted that an increase in NaOH concentration led to an increase in open volumes (or open porosity) or defects in the zeolite structure. On the other hand, increasing the temperature during sample preparation tended to reduce or diminish the open volumes, indicating a change in the zeolite's structural characteristics [101]. The direct hydrothermal method is a straightforward process, but it has drawbacks in terms of time and energy consumption, leading to low product purity [102]. Also, the presence of contaminations in FA like, for example calcium oxides, limits the purity of the final product [103]. To overcome these limitations, alternative techniques like alkaline fusion-assisted and microwave-assisted hydrothermal methods have been introduced to enhance the yield and quality of zeolites. The advantage of using microwave-assisted over alkaline fusion-assisted hydrothermal methods lies in the increased crystallization rate, leading to reduced crystallization time [9]. In turn, the use of microwave synthesis allows for high control of the temperature and time of crystallization, which is essential in obtaining specific crystalline phases. Additional extraction of silicon from FA allows us to achieve zeolites of high quality and purity. The desired Si/Al molar ratio might be obtained by adding aluminum ions [52]. The more frequently used method is also hydrothermal alkaline fusion, in which a solid mixture of NaOH with FA is used, which is then heated to a temperature of about 500–600 °C [104]. However, due to high energy consumption the fusion method is rather expensive and the production line on the technical or industrial scale is not economically justified [105]. By employing ultrasonic energy, the aging time for the alkaline fusion followed by the ultrasonic-assisted synthesis method is significantly reduced from 24 to 2 h. This ultrasonic aging process allows for an approximately 90% reduction in aging time and related costs, primarily attributed to the decrease in electricity consumption [106].

More frequently, combinations of methods are also used to enhance the quality and properties of the final product. Just recently, for the first time, researchers have successfully synthesized a highly crystalline zeolite from CFA using a unique approach that combines in situ microwave and ultrasound activation. This method is known as microwave and ultrasound collaborative activation (MUCA) and it gives better results than individual activation methods using microwave or ultrasound alone. MUCA exhibits higher efficiency in dissolving silica-aluminum species from CFA, while preserving other impurities and avoiding their digestion. As a result, MUCA significantly reduces energy consumption during the zeolite synthesis process [107].

In addition to the potential reduction in energy consumption during zeolite synthesis, a high-purity zeolite has been successfully produced from FA using a green synthesis approach. This environmentally friendly method proposed in [108] involves the minimal use of water, with only trace amounts utilized, and it achieves zero discharge of wastewater during the synthesis process [108]. A CFA-derived zeolite might also be produced using another new and convenient solvent-free method, ensuring minimal energy consumption due to its short processing time as described in [109].

Recent findings also prove that the particle size distribution of utilized FA significantly influences the quality and yield of the obtained zeolites. By incorporating fine FA particles, a larger reaction area might be achieved, resulting in the formation of zeolite phases with a higher degree of crystal formation, increased purity, and enhanced crystallinity. These improvements in the physicochemical properties directly correlate with a considerable improvement in their potential applications. Additionally, this can be achieved with only a relatively small financial investment required for the FA grinding process [110].

In [111], an additional step of wet grinding was proposed to enhance the synthesis process of zeolites from FA. During grinding, the raw material undergoes destructive forces such as friction, shear force, and impact, leading to a decrease in particle size. The process generates heat, which promotes the activation process of FA. The key parameters affecting the particle size change and system temperature increase are grinding time, grinding speed, ball–powder ratio, and solid–liquid ratio [111]. Grinding has the advantageous effect of reducing particle size, increasing the surface area, and enhancing the surface structure of coal-based solid wastes. These improvements are beneficial as they facilitate better exposure and subsequent removal of impurities [112]. It was noted that, when the wet-grinding time increases, the particle size gradually decreases until reaching stability after 2 h. During wet-grinding, the raw material develops structural defects, but with prolonged grinding, these defects decrease, and particle strength increases. Consequently, the crushing difficulty increases, making 2 h the optimal grinding time. Using FA as a raw material for zeolite synthesis through the wet-grinding activation hydrothermal method reduces energy consumption and the number of process steps. This approach holds the potential for the large-scale use of FA to create an inexpensive adsorbent, making it a promising option for practical application [111].

The incorporation of mesopores into microporous zeolites, known as hierarchical zeolite synthesis, represents a highly promising method to enhance the surface properties and porous structure of zeolites. When the diffusion of ions and molecules to an active site is limited, the internal surface area of a zeolite remains empty, leading to reduced zeolite activity. However, this challenge can be addressed by synthesizing hierarchical zeolites that possess enhanced diffusion capabilities [99]. This approach combines the benefits of shape selectivity from the microporous framework with the facilitated mass transfer achieved through the mesoporous channels [113]. Hierarchical zeolites, possessing a dual-porous structure, have emerged as a significant and novel category within the field of zeolites. Their potential lies in their ability to enhance mass transfer and molecular accessibility, crucial for overcoming obstacles such as steric hindrance, diffusional constraints, and coke formation in catalytic reactions [114].

Numerous techniques have been developed to synthesize hierarchical zeolites, including organosilane-based procedures, surfactant-assisted methods, framework atom removal, zeolite synthesis of preformed solids, templating, and the modification of porous materials like activated carbon and diatomite. However, these methods are expensive due to their multi-step processes involving templates and acid-base treatment after synthesis. Alternatively, hierarchical zeolites can be synthesized in a cost-effective and straightforward manner using the microwave heating method [99].

The technological production line described in [115] generates very good results regarding high-purity Na-P1 zeolite synthesis. Most pilot-scale facilities are conducting the conversion process of FA with NaOH at a moderate temperature at atmospheric [115] or elevated pressure [116]. The obtained zeolites were tested as water treatment adsorbents for capturing heavy metals and ammonium [116]. Whereas zeolites obtained via fusion followed by a bench-scale hydrothermal reaction and hydrothermal pilot-scale reaction were analyzed as potential fertilizer carriers [117].

4.3. Standard Characterization Methods of Zeolite Materials

Zeolite materials can be characterized using various analytical methods to gain insights into their structure, composition, porosity, surface properties, and other relevant parameters, which depend upon their intended function.

Some of the common methods of characterization for zeolites include:

1. X-ray Diffraction (XRD) is used to determine the crystalline structure and phase composition of zeolites. It provides information about the arrangement of atoms in the zeolite framework and helps identify different zeolite phases [118]. It is a valuable tool in the determination of the efficiency of synthetic zeolite synthesis methods [7].

X-ray diffraction (XRD) analysis can be utilized to estimate the crystallinity degree of a specific zeolite. This calculation involves comparing the intensities of the characteristic zeolite profile with those of the reference materials, as expressed in the following equation:

$$Cristallinity(I/I_0) = \frac{\sum relative\ intensities\ of\ fly\ ash - derived\ zeolite}{\sum relative\ intensities\ of\ a\ reference\ material} \times 100 \quad (1)$$

2. Scanning Electron Microscopy allows us to visualize the surface morphology of zeolite materials at a high resolution. It provides information about particle size, shape, and distribution. Additional EDS (Energy-Dispersive X-ray Spectroscopy) gives information about the semiquantitative chemical composition of the material. Observation of the sample morphology enables an estimation of its porous structure development through synthesis procedures. SEM has been used to investigate zeolites' microstructure, particle morphology, and crystal growth [115,119,120].
3. Transmission Electron Microscopy (TEM) offers a higher resolution than SEM and allows us to examine the internal structure of zeolite crystals at atomic resolution, including the arrangement of atoms and crystal defects [121–123].
4. Low-Temperature Gas Adsorption using nitrogen or carbon dioxide (BET Analysis) is used to determine the specific surface area and porosity of zeolites [9,18,124]. It provides information about the structure of the material and its potential sorption capacity. To more precisely determine the sorption properties of the material toward a given adsorbate or contamination more advanced methods are needed, like, for example the dynamic vapor sorption method (DVS) [125].
5. Fourier Transform Infrared Spectroscopy (FTIR) is used to identify the chemical bonds and functional groups present in zeolite materials, providing information about their composition and surface properties [118,126].
6. Thermogravimetric Analysis (TGA) with Differential Scanning Calorimetry (DSC) together with Simultaneous Thermal Analysis (STA) is used to study the thermal stability of zeolites and to determine the water content and their desorption behavior. TGA and DSC are used to determine the thermal properties of zeolites, including phase transitions and thermal stability. These properties are crucial when zeolites are dedicated to high-temperature purposes like flue gas treatment. A unique method based on TGA was also proposed in [127], dedicated to determining the degree of fly ash conversion efficiency.
7. Nuclear Magnetic Resonance (NMR) Spectroscopy can provide information about the local environment of certain atoms in the zeolite structure, giving insights into their connectivity and coordination [126]. Due to its remarkable sensitivity to the atomic-scale environment, this method is well-suited for investigating local structure, disorder, and chemical reactivity in solid-state materials [128].
8. X-ray Photoelectron Spectroscopy (XPS) is used in some cases to study the chemical composition and oxidation states of the surface of zeolite materials [129].

By combining these characterization methods, research groups working in the field of zeolite synthesis can gain a comprehensive understanding of zeolite materials and optimize their synthesis methods and use in various applications.

4.4. FA-Derived Zeolites as Value-Added Products in Different Industrial Branches According to the CE Concept

In general, zeolites are characterized by a very large specific surface area, due to which they can be used as sorbents, catalysts, and ion exchange materials [130–132]. In nature, zeolites are represented as minerals (e.g., clinoptilolite, chabazite, and mordenite). The type of zeolites depends on the SiO_2 content (SiO_2/Al_2O_3 molar ratio), therefore they can be classified as low-, medium-, and high-silica zeolites. The Si/Al ratio determines the properties of the zeolite. Low-silica zeolites are characterized by higher acid resistance, stability at higher temperatures and hydrophilicity. On the other hand, high-silica zeolites are more hydrophobic and have high ion-exchange properties. The properties of natural zeolites in aqueous solution depend on the pH; a very low pH value can cause damage to the structure of the zeolite. Zeolites of the NaP1 type, A type, X type, Y type, and ZSM-5 zeolite are the most frequently studied and used in industry [89,133,134].

Currently, synthetic zeolites are successfully used in a wide range in industry and environmental protection, for example as soil-improving additives and as adsorbents in water and gas purification systems (Figure 5). FA-derived zeolites X and A might be employed to capture elemental mercury from a gas stream. The proper capture and immobilization of mercury is especially important as large EU combustion plants (LCP) are obliged to monitor and minimize the emission of this contaminant from the flue gas stream [135]. A unique hybrid synthesis approach was developed, incorporating the crystallization stage of zeolites, along with silver or iron modification. This method successfully yielded pure and high-crystalline zeolites. Notably, it was observed that silver-modified zeolites exhibited significant potential in adsorbing mercury [136]. The zeolites synthesized from coal FA (CFA) were also effectively employed to remove methylene blue [119] and Cu ions from aqueous solutions [137].

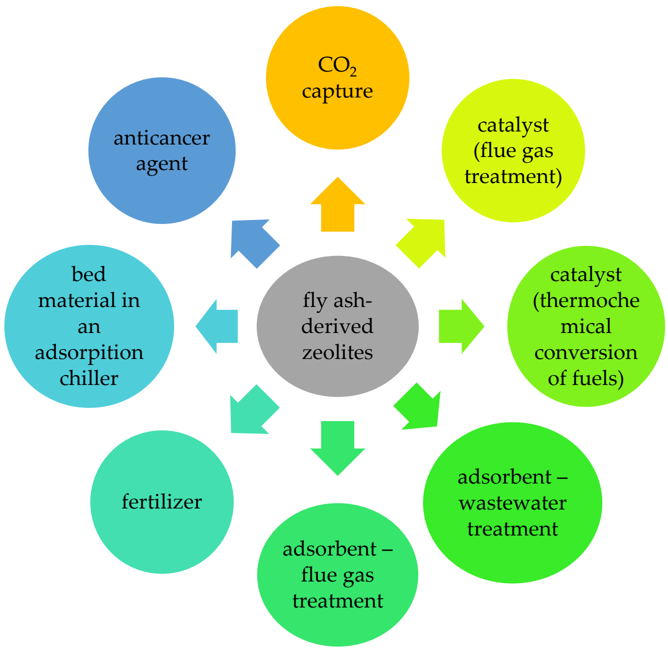


Figure 5. FA-derived zeolites' utilization in industry, agriculture, and power sectors.

As for the more specific use of zeolites synthesized from FA, they can also be used in adsorption systems for CO_2 [138,139]. It has been observed that a higher calcium content in a FA positively affects CO_2 retention under equilibrium conditions. However, this effect is not as significant in dynamic conditions, likely due to the presence of accompanying chemisorption processes [140]. Zeolites also serve as great adsorbents in gas treatment facilities, capturing contaminations [97] like nickel (Ni) [141], arsenic (As) [142], zinc (Zn), copper (Cu) [143], and lead (Pb) [111,144]. Zeolites are also used as a bed material in adsorption refrigerators [145–147], because they are sorption materials characterized by excellent sorption properties: high adsorption capacity, molecular shape selectivity, and a very large surface area. A synthesized zeolite may be characterized by superior properties compared to a commercial zeolite, making it a more appealing choice for gas separation applications. Notably, in some cases synthesized zeolite displayed a higher surface area, smaller crystallite size, and improved thermal stability, all of which contribute to its enhanced performance in this specific context [148,149]. Zeolites might be also used as catalysts in gas treatment systems. ZSM-5 zeolite obtained from CFA was used as a catalytic converter for the reduction of NOx in gasoline-powered engines. By incorporating copper and cobalt-doped zeolite on a ceramic monolith, the catalytic converter demonstrates superior activity in rapidly reducing NOx emissions compared to conventional catalytic converters [150]. Another CFA-based ZSM-5 zeolite characterized by a flexible textural design and obtained using a solvent-free method gave a good result for toluene adsorption elimination [151].

Additionally, zeolites are characterized by a three times greater water adsorption capacity compared to silica gel, and the artificial synthesis of these materials allows for improvement of the efficiency of heat exchange, making them a good adsorbent for adsorption chillers [152,153]. A certain limitation to the use of zeolites in refrigeration is the fact that the bed regeneration process is carried out at temperatures higher than in the case of silica gels, i.e., above 120 °C [154]. In addition, as in the case of other porous materials, zeolites are characterized by a low coefficient of thermal conductivity [155–157]. However, the high sorption capacity and durability of these materials mean that commercial solutions based on a zeolite bed are available on the market.

The recycling of coal FA offers a promising opportunity to create highly efficient Fenton-like catalysts capable of removing organic contaminants from wastewater effectively [118]. An additional product of synthesis might include a magnetic type which could be used as a substrate in nano-iron synthesis [109].

FA-derived zeolites hold potential not only as adsorbers in various industrial processes but also as valuable new products or components. For instance, the treatment of FAs significantly influenced both the synthesis yield and properties of the resulting zeolites. A well-designed zeolite synthesis method effectively reduced the viscosity of modified asphalt cement during heating, greatly improving its workability. The observed excellent performance of zeolite A, derived from FA, in reducing asphalt cement viscosity makes it well-suited for this specific purpose [158].

Fertilizing zeolites have demonstrated significant potential in providing fertilizer elements over extended periods of time, making them a suitable solution for more demanding crops' nutrient requirements. As a result, these synthesized materials effectively serve their purpose as slow-release nutrient suppliers, showing promising applications as K fertilizers. Moreover, a cost-effective method of FA utilization in producing a zeolitic product with a well-structured framework capable of efficiently retaining K ions was proposed. This product, called MER zeolite, exhibits improved performance in releasing K nutrients, thus minimizing nutrient wastage and reducing the risk of environmental pollution [159].

Prior to disposal in landfills, municipal solid waste incineration fly ash (MSWIFA) with elevated heavy metal concentrations requires consolidation. The alkali-activated technique plays a crucial role in enhancing the degree of heavy metal solidification and the utilization ratio on MSWIFA material. Incorporating zeolite into the process shows promising results, as it increases the efficiency of heavy metal solidification within the FA matrix, making it a potentially valuable application of FA [160].

Self-supporting adsorbents composed of zeolite-geopolymer composites, synthesized using industrial solid waste, exhibit remarkable mechanical strength, a high specific surface area, and cation exchange capability. These composites serve as effective substitutes for natural heulandite zeolite in removing metal ions from water. The simple two-step method allows for the synthesis of zeolite–geopolymer composites with a high zeolite content, making them highly suitable as self-supporting adsorbents for water treatment applications [81].

An effective method for defluorination of water involved the use of aluminum hydroxide-coated zeolite (AHZ), which was synthesized from coal FA. AHZ proves to be efficient in removing fluoride from water and offers a promising solution for addressing fluoride contamination issues in wastewater treatment [161]. The synthesized zeolite can be then effectively employed as an adsorbent to eliminate organic dye from aqueous solutions as well [162].

The zeolites obtained from the coal FA were also subjected to testing in tgas-phase phenol alkylation using diethyl carbonate (DEC) as a novel alkylating agent. The results from the experiments demonstrate the practicality and suitability of obtained innovative synthetic zeolites for catalytic applications as heterogeneous basic systems. The method employed yielded high conversion rates, was environmentally friendly, cost-effective, and presented a clean approach. Consequently, the synthetic zeolites proved to be highly efficient catalyst alternatives to the commonly used commercial materials in industrial applications [163].

In Ref. [164], an investigation was conducted on the mineral and chemical compositions of FA to determine its suitability as a source material for synthesizing zeolites, which serve as useful matrices for nuclear waste vitrification. Additionally, the findings in [2] indicate that K-zeolites synthesized from biomass FA are well-suited for the removal of radioactive cesium. Biomass-origin FA is characterized by a low content of impurities making it possible to exclude additional purification from the zeolite synthesis procedure [165]. Additionally, this process offers a potential solution for recycling FA from biomass power plants, thus avoiding the high economic costs associated with its disposal.

Zeolites might be also used as catalysts in thermochemical fuel conversion processes [95,132,166]. FA-derived zeolite X was used as a suitable catalyst for the production of linear aliphatic hydrocarbons (HC), although some concerns have arisen due to the deactivation caused by coke formation. The data obtained from research conducted in [167] can be applied to enhance the quality of bio-oil produced from local biomass resources, pyrolyzing palm press fiber (PPW), through catalytic fast pyrolysis.

The groundbreaking experiments conducted on zeolite X synthesized from coal fly ash have revealed its potential as a significant anticancer agent. However, further research is still necessary to understand the specific mode of action [149].

To sum up the recent findings regarding FA-derived zeolites, the properties of the obtained materials are presented in Table 3.

Table 3. Comparison of specific surface area and pore diameter values for fly ash-derived zeolite materials.

Adsorbent	Specific Surface Area	Pore Size	Reference
Unit	**[m^2/g]**	**[nm]**	**-**
Na-X zeolite obtained from FA from oil shale combustion (China)	252	3.817	[167]
Na-P1 zeolite obtained from FA from lignite combustion (Greece)	64.5	0.8	[168]
NaA zeolite obtained from CFA (South Africa)	33	-	[139]
NaX zeolite obtained from CFA (South Africa)	228	-	[139]
Na-P1 zeolite obtained from CFA (India)	63	-	[169]
Zeolite ZSM-22 obtained from CFA (Taiwan)	30.2	-	[170]
Zeolite X obtained from CFA (Poland)	629	1.7	[171]
Zeolite NaP1 obtained from FA from the waste incineration process	23.86	-	[172]

Table 3. *Cont.*

Adsorbent	Specific Surface Area	Pore Size	Reference
Unit	**[m^2/g]**	**[nm]**	**-**
FA-derived zeolite NaP1 from class F FA, obtained from a coal power plant (Poland)	60	11	[143]
Na-P1 zeolite obtained from CFA (Poland)	88	-	[115]
Sodalite zeolites obtained from CFA (South Africa)	366–399	-	[109]
Zeolite-geopolymer composites obtained from FA and metakaolin (China)	52–100	4–7	[81]
Zeolites-calcium silicate hydrate composite obtained from CFA (China)	96.5	-	[103]
Zeolites obtained from CFA collected from the electrostatic precipitators (Bulgaria)	284–486	13.2–61.1	[140]
Zeolite X prepared from FA (China)	473.56	1.9	[108]
ZSM5 obtained from CFA (Thailand)	329	6.0–27.8	[173]
X zeolite obtained from CFA (Thailand)	722	5.7–24.2	[173]
Zeolite 4A obtained from CFA (China)	18.33	11.3	[137]
Zeolite 4A obtained from CFA (Pakistan)	122	-	[148]

Zeolites synthesized from fly ashes have a significantly lower specific surface area compared to natural and commercial zeolites (Table 2), which is due, among other things, to the fact that fly ashes are not a pure mixture of silicon and aluminum compounds. They also have other impurities that affect the content of the zeolite phase obtained in the final product. In addition, the final product depends to a large extent on the method of synthesis and its parameters. FA preparation in order to obtain native material more susceptible to the synthesis of zeolite material, such as silicon extraction, significantly increases the cost of synthesis.

However, the synthesis of zeolites with coal-based solid waste as a raw material can reduce the environmental pollution of industrial wastes, minimize natural resource utilization, and realize their valuable utilization in line with the CE concept.

5. Conclusions and Future Directions

Recent research findings present fly ash-derived zeolites as promising materials that subscribe to the principles of CE by turning a waste stream into a valuable resource. Their diverse applications in environmental remediation, catalysis, material substitution, and medicine demonstrate their potential to implement sustainable practices and contribute to a more circular and resource-efficient future. The main challenges are associated with synthesis by-products, especially additional waste generation and energy consumption. Furthermore, in not all methods can high purity of the final product be achieved. FA contaminated with other elements like heavy metals and calcium might not be suitable for zeolite synthesis. High energy consumption in the case of hydrothermal methods at a high temperature makes them economically unjustified in a larger scale.

Further research and collaboration between researchers, industrial stakeholders, and policymakers are essential to fully realize the benefits of these materials and their integration into CE practices. However, utilization of FA-derived zeolites requires the precisely defined chemical composition and properties of raw FA, as synthesis methods are adjusted to the given feedstock.

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