Review

Recent Advances in Methods for Recovery of Cenospheres from Fly Ash and Their Emerging Applications in Ceramics, Composites, Polymers and Environmental Cleanup

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Abstract: Coal fly ash (CFA) is a major global pollutant produced by thermal power plants during the generation of electricity. A significant amount of coal fly ash is dumped every year in the near vicinity of the thermal power plants, resulting in the spoilage of agricultural land. CFA has numerous value-added structural elements, such as cenospheres, plerospheres, ferrospheres, and carbon particles. Cenospheres are spherical-shaped solid-filled particles, formed during the combustion of coal in thermal power plants. They are lightweight, have high mechanical strength, and are rich in Al-Si particles. Due to cenospheres' low weight and high mechanical strength, they are widely used as ceramic/nanoceramics material, fireproofing material, and in nanocomposites. They are also used directly, or after functionalization, as an adsorbent for environmental cleanup—especially for the removal of organic and inorganic contaminants from wastewater. By utilizing this waste material as an adsorbent, the whole process becomes economical and eco-friendly. In this review, we have highlighted the latest advances in the cenospheres recovery from fly ash and their application in ceramics and wastewater treatment.

Keywords: coal fly ash; ceramics; cenospheres; alumino-silicate; ferrospheres

1. Introduction

Coal fly ash (CFA) is one of the major pollutants of the 20th and 21st centuries. It has drawn global attention regarding its safe management, optimized generation, and utilization [1]. Globally, a million tons of CFA are generated per year, in thermal power plants (TPPs), from the burning of pulverized coal, during the production of electricity [2]. The huge amount of generation of CFA leads to air, water, and soil pollution. Every year, a huge amount of CFA is dumped in the near vicinity of the TPPs, in fly ash

ponds, which ultimately leads to water and land pollution. These fly ashes contain heavy metals, which can leach out once they contact water [3]. Besides heavy metals, CFA also contains minerals, such as magnetite, calcite, mullite [4], cristobalite, and silica [2]. These minerals contribute to the silica (40–60%), alumina (20–40%), and ferrous (5–15%) that rely on the source and types of coal used [2].

Since TPP furnaces are operated at very high temperatures (1000–1800 °C), the organic matter of coal becomes decomposed into a molten slag, which attains morphologically variable shape and size [5]. This variation leads to structurally different types of particles in CFA, namely, plerospheres, ferrospheres, and cenospheres (CS) [6]. The plerospheres are encapsulated particles that have numerous smaller particles within them, along with gases and minerals [7]. While, as the name suggests, ferrospheres are ferrous-rich spheres or "spherical particles" [8], and cenospheres are Al-Si rich spheres or "spherical particles" [2]. These Al-Si-rich particles are also known as aluminosilicate spheres or CFA microspheres [2]. As well as these spherical particles, there are varying amounts of irregular-shaped organic and inorganic carbon particles, i.e., unburned carbon soot and chars.

Among all the spherical particles, CS have been a focus in the field of ceramics, construction, lightweight materials, agriculture, etc., due to their unique physical and mechanical features. The word "cenospheres" is derived from the two Greek words—cenos, meaning "hollow", and spahira, meaning "spheres" [9]. These are spherical-shaped, mullite-rich [4,10], inert, thin-walled hollow ceramic particles that have several trapped gases within them [11–15]. Their size falls in the range of 1–600 microns, and their total wt. fractions is about 1–2% in CFA [16]. From the literature, it is known that the water absorption capacity of porous CS is about 18 times higher than that of sand [6,17]. The hollow nature of CS is due to the presence of trapped gases [18]. Due to the hollow nature of CS, their density is near 2.3 g/cm^3, whereas the buoyancy of a CS is provided by its density. The bulk density of CS is around 400 kg/m^3 [19,20], and the true density is ca. 2.3 g/cm^3 [21]. Their high mechanical strength, high porosity, and thermal shock-resistance have encouraged scientists to use them in ceramics, the preparation of nanocomposites (decorating micron-sized CS with nanomaterials), and for environmental cleanup [22].

This review emphasizes the morphological, chemical, and physical properties of fly ash cenospheres. The purpose of this work was: (1) To provide all the methods applied, to date, for the recovery of cenospheres from coal fly ash, and (2) to reveal the recovery efficiency with all the applied methods. This work highlights the well-known and emerging applications of cenospheres. The main objective of this review was to provide state-of-art applications in the field of environmental cleanup and in ceramics and constructions.

2. Morphological Properties of CFA Extracted Cenospheres

Based on the morphology, viscosity of liquid composition of slag, and elemental composition, some investigators have classified the CS into two broad types—namely, nonmagnetic CS and magnetic CS. The formation of magnetic CS depend upon the melts of A-Si and Fe-Al-Si [9]. High viscosity type cenospheric particles have more tendency to trap gases and increase in diameter of the particle, resulting in the thinner shell with the complete absence of pores [23]. Whereas, in the case of a less viscous cenospheric material, the hot flue gas can pass through the material, due to the decreased viscosity. As a result, there are fewer chances of trapping the gases; hence, the diameter/shell thickness reduces to about 3–4 with a porous shell structure [23].

Certain cenospheres have high Fe deposition on their surface that exhibit magnetic properties, while others are mainly rich in Al and Si (along with Na and Ca). In numerous pieces of literature, CS with small Fe deposition have been widely in the petroleum cracking, catalysis, ceramic, nanocomposites, and steel or iron-based industries [2]. Several investigators have considered the Fe deposition on CS as an important property, as based on this, only both nonmagnetic and magnetic CS can be identified and differentiated very easily [9], and widely used in the petroleum cracking, catalysis, ceramic, nanocomposites,

and steel or iron-based industries [9]. Based on some physical parameters, i.e., texture, shell thickness, and shape, cenospheric particles are either transparent, grey, and dark type [24]. The former type of particle is thin-shelled, smooth-surfaced, grey type are translucent, porous, thick, and rough-surfaced [9]. While the dark-type CS have numerous perforations on their surface, their porosity is much greater than the grey CS. Dark CS scatters the light completely, which is passed through them and acts as an opaque particle. Ferrous-rich CS can be divided into two types: One having a porous shell, while the other with a spotty nonuniform surface, containing grey, black, brown, and white inclusions, along with dark black or brown spheres with a bright and smooth surface [25]. The Al content contributes a valuable role in the average size of a nonmagnetic CS, i.e., more Al equals higher mullite, and ultimately results in a decrease in the average diameter [9,26,27].

CS can be divided into two main groups, based on their magnetic features, which are acquired by the presence of Fe_2O_3/Fe_3O_4 minerals in the shell [28]. The shell of CS is a complex, layered structure (which is covered by a nano-size film externally), and internally [25,29] (which is 30–50 nm thick and contains Fe_2O_3). When the ferrous oxide percentage varies from 3 and 4 wt.% in the alumino-silicate glass, then two types of Fe^{3+} are present, i.e., single ions and nanoparticles with a diameter of 3 to 5 nm. These particles consist of a superparamagnetic phase with a spinel structure whose sublattices are diamagnetically diluted with Mg^{2+} and Al^{3+} ions. CS, having about 7 wt.% of Fe_2O_3 content, also involves a magnetic phase, based on defective magnetite [30].

Magnetic CS have heterogeneous regions of ferrospinels on their outer surface. An increase in the concentration of iron increases the crystallite size of the ferrospinel phase in the magnetic CS, and decreases the degree of iron substitution (Mg and Al) [9,26]. In magnetic CS, shell thickness is independent of the shell composition. When the Fe_2O_3 content in magnetic particles (having porous shells and close average diameter values) increases by 4 to 21 wt.%, it causes an increment in the number of spotty spheres (having a nonuniform surface) by 13–81 volume % and in the number of dark globules (having smooth surface) up to 26 volume % [25].

In general, CS size falls in the range of 20–300 microns, with a shell thickness of 1–18 microns, but the average size is 5–500 microns [31]. In comparison to the CFA particles, CS are much larger, and can vary from 5 to 500 μm [15]. Ranjbar et al., 2017, said that the shell thickness of CS increases linearly with the increase in the diameter of the CS particle [9]. Ngu et al., 2007, reported that the ratios between wall thickness and diameter of ash cenospheres are limited between an upper bound of ∼10.5% and a lower bound of ∼2.5%, irrespective of the ash cenosphere size [10]. When the coal is present in the molten slag, then the size of the cenosphere particle is governed by the temperature of the furnace of TPPs [13]. At a higher temperature, the molten slag of the coal is blustered into numerous smaller fragments, which are observed after the solidification of the cenosphere particles. Besides temperature, the particle size of a cenosphere in a TPPs is also governed by the chemical composition of the molten drop [13]. Several studies from the literature revealed that as the particle size of a cenosphere increases, Si and Al content decreases— but there is a larger decrease in Si, than the Al in the cenosphere because of the larger amount of Si present. Consequently, the alumina/silica ratio increases. As the particle size increases, then the Al content also increases. Due to the increase in Al content, the viscosity of the molten drop also increases, which stabilizes the large drop particles and prevents them from exploding into various smaller fragments. If particle size increases, then the alumina/silica ratio also increases, or if particle size decreases, the alumina/silica ratio decreases [15].

The formation of CS from pulverized coal is a very complex process that depends on the mineral matter, composition of coal, and combustion parameters of thermal power plants (TPPs) [12,14]. The cenosphere formation mechanism is similar to a glass blowing process. Firstly, coal is converted via molten slag at high temperatures, then secondly, molten slag starts flowing in the gas stream and undergoes rapid quenching [23]. Due to this rapid quenching, cooled molten slag falls into the formation of the sphere. After

quenching, the particles are transformed into spheroids or spheres, and the minerals and gases present within them or in the nearby area become trapped inside the particles. These types of bubbles are responsible for the formation of CS, and could be either in multiple forms within the frozen particles or in single concentric forms—whose size in diameter is equivalent to the particles [20]. Because these particles interact for a very short time along with the influence of wettability parameters, numerous particles merge or coalesces into a large particle by maintaining the silicate and ferrous melted composition relationship between the two [20]. The formation of CS from pulverized coal in a TPP is justified by two hypotheses: One hypothesis suggests that condensed gases are formed because of the breakdown of the various organic and inorganic constituents of coal; thus, there is the evaporation of pore water in coal that becomes trapped in molten microspheres [20]. These trapped gases inflate the molten drop, increasing the size of the particles up to 500 μm [13,15], and resulting in a spherical- and hollow-shaped particle. Whereas, the second hypothesis suggests that inside the furnace of TPPs, there are high-temperature zones where the molten ash droplets are first formed while being penetrated by the hot flue gas [23]. Once these particles are cooled down, these gases remain inside the spheres, which are called CS.

3. Properties of Cenospheres Recovered from CFA

Structurally, cenospheres are hollow spherical-shaped particles that resemble glass beads, micron-balloons, or hollow ceramic micron-sized spheres [32]. Their size is 5–500 microns [13], and an average size is 30 μm to about 350 μm [10,15]. The cenospheric weight in a CFA comprises approximately 0.1–4.8% of the total weight of CFA [25], as shown in the literature.

There might be slight variation in the weight fractions from different parts of the world. As far as chemical compositions are concerned, CS contains elements, such as Ca, Mg, K, Na, Ti, Al, Mg, Si, Fe, C, and O [13], which may be present as different mineral phases [33]. Moreover, several investigators have also reported a trace of elements like V, Zr, Ba, Sr, S, P, and Rb. Strzałkowska and Adamczyk, 2019, reported all the above-mentioned elements in the CS recovered from three different TPPs (Dolna Odra, Opole, and Kazakhstan) [13]. Furthermore, Danish and Mosaberpanah, 2020, analyzed the chemical composition of CFA from 12 different TPPs, from various parts of the world, and found that all of them have Si, Al, and Fe as major elements, while Ti, Ca, Mg, Na, S, and Mg were present in smaller quantities [14]. The common elements were Al, Si, Fe, O, and Ca, whose composition may vary, but the presence of the other elements depend on the type of coal, source of coal, geographical origin, and furnace temperature during the burning of coal in the TPPs. When water or rain comes in contact with these toxic elements, it may leach out from there and may have a negative effect on the material [34,35].

Since CS have two surfaces, i.e., inner and outer, which are covered by a nano-thin film whose thickness is between 30–50 nm [30]. The inner surface is mainly composed of a glass of alumina and silica, and the skeleton is comprised of crystalline minerals like quartz, mullite, and magnetite [16]. Both of these surfaces make a shell composed of a crystalline phase and amorphous glass phase. The thickness of the crystalline phase is 2–30 microns, while for the amorphous glass phase, it is 50–90 microns [25]. The major crystalline phase is dominated by mullite, magnetite, calcite, cristobalite, feldspar, and hematite [36], which play a role in the morphology and elemental composition of CS. The average gas pressure inside a CS is below one atm within the range of 0.172–0.227 atm, which decreased in larger particles, since more gases would be required for expansion [37,38]. The stability of the hollow spheres is dependent on the equilibrium of surface tension forces on melt drops and intrinsic pressure of the internal hot gas [23]. The CS are bestowed with numerous physical properties like lightweight, low thermal conductivity (TC) [39], thermal shock-resistant [39], ultra-low density [31], highly porous [40,41], fire-proof [42], resistant to acid and bases [43], resistant to oxidation and corrosion [44], excellent mechanical strength [10], and protected from electromagnetic interferences [9,45–48]. The thermal shock-resistant properties in

the CS are added from the mullite content [49], which is chemically inert. Furthermore, the porous nature of the CS enhances their water absorption capacity, thus it is considered a potential candidate as an absorbent for wastewater treatment and environmental cleanup [50].

4. Methods Used to Recovery Cenospheres from CFA

The recovery of CS from CFA is possible by both dry- and wet-based methods [51]. As the name suggests, the wet method involves a liquid media like water and or any organic solvent, whereas the dry-based method utilizes either air stream or size-based screening. Among all the techniques for recovery of CS from CFA most common methods are magnetic separation, sedimentation, flotation, or sink float method [21]. All these recovery methods are carried out in a separation tank which contains water (1 g/cc) and acetone (0.789 g/cc), which is fitted with agitation or stirring, and consequently, heavier particles settle down, while lighter particles float at the top. The dry-based method mainly uses air classifiers to separate CS particles [21,52].

4.1. Wet Separation

The sink float process is the most common method to separate the different components of CFA, followed by the gravity separation [53], are provided below in detail. Both of these methods involve a liquid medium, such as water and acetone, to separate the CS [54]. Manocha et al. reported the collection of four types of fly ashes from different power stations from the Gujarat region in India. They applied the sink flotation method for the separation of CS from all these four samples of fly ashes by using two different types of solvents having different densities. The recovery method helped in the extraction of about only 1% of the total composition of CS from the fly ashes by weight fractions [48].

The gravity separation method is one of the most preferred methods for the recovery of CS from CFA, due to its simplicity and common availability [55]. Since it is a wet-based method, it involves using liquid phases, such as water, acetone, etc., and works on hydraulic separation technique [21], whereas because is CS lighter in weight, it floats over the suspension. Furthermore, these suspended CS are collected by the pond skimming method [52,56]. Since this technique relies on the principle of physical parameters (density of solid and liquid particles, feed particle amount, buoyancy, porosity, and texture of the particles), so the efficiency of recovery of CS from CFA depends on all these factors. It could be better explained, for instance, a smooth surface particle will float faster than rough-surfaced particles, as smooth particles will have lesser interaction with the liquids, surfaces, and refinement cycles.

Beddu et al., 2021, used the sink-float technique to extract CS from CFA obtained from Malaysian thermal-based power plants. They designed a special rectangular reactor composed of acrylic. Here, the CS were extracted from CFA using the wet method (floatation/float and sink) [52].

Extraction of CS from lignite-based CFA (class C from Thailand) by sink-float technique was reported by Yoriya et al., 2019 [15]. Since his method involves a physical-method approach, a better quality of CS was achieved. This method was a wet separation method, where the used solvent was a mixture of acetone–water. Furthermore, the investigator used different ratios of water and acetone and observed the effect on the yield, morphology, and elemental composition of CS. They also concluded that calcium carbonate and CaO affected the recovery yield of CS. When the water-acetone ratio was (0–50%), then the yield was significantly less, as the mixture had a lower density. Furthermore, when the liquid density was >0.90 g/cc, then the CS yield also increased significantly. With the increase in acetone content, the density of the medium lowered, resulting in CS size of larger size. The Si and Al content gradually decreases with the decrease in the acetone content. The obtained CS was spherical in shape, while the CS obtained with high acetone (80% and 100%) have a clean surface. The recovery by this technique was reported to increase the separation efficiency, recovery yield, and quality of CS [15].

Petrus et al., 2011, used a float-sink test for the dry separation method, based on extracted CS. Their intention was to determine the quantity of cenospheric particles in the overflow and underflow products [57]. Liu et al. also extracted the CFA CS by using the flotation method. They used ethanol over here in the first step [58]. Alcala et al., 1987, reported the extraction of CFA CS from CFA by two different methods, i.e., flotation and simple sedimentation. Flotation experiments were carried out by using four different frothers as unique reagent addition. The buoyancy flotation technique (BFT) is based on the density of the CS. Therefore, cenospheres are usually concentrated using their natural buoyancy in water. Ali Kiani, 2016, reported the recovery of CS from CFA from various thermal power plants of Australia [59].

There are several reports where the skimming technique was applied for the recovery of CS from CFA. Two US patents, namely, US Patent No. 4121945A (1978) [60] and US Patent No. 4,652,433 (1987) [61], used the skimming technique. This technique mixes CFA with water to form a slurry, and then adds a frother, resulting in CS that is lighter than the skimmed water. However, both the patents reported certain drawbacks associated with this skimming technique. For instance, space, environmental concern, not fit for high lime containing CFA was some of the major ones. The advantages of this technique are that it can yield nearly 100% CS, and it avoids the agglomeration of CFA having high Ca content [62].

The efficiency of the wet separation techniques can also be increased by the clubbing of two or more different separation approaches like hydrodynamic gravitational separation, grain-size classification, and magnetic separation [15,21]. One of the most important advantages of wet separation is that by altering the recovery process, it is possible to separate destroyed CS. The wet-based method has numerous disadvantages—for instance, trace elements of CFA are dissolved in the medium, which can leach toxic heavy metals that are fatal for living beings [21]. Land and water are the basic requirements of this process which is not economical. Due to the presence of high calcium content in CS, it has restricted uses in cement or in cement additives. In this process, the recovered CS are wet and must be dried, which is an energy-intensive process, and hence, expensive.

Kiani et al., 2016, used a unique and innovative novel system to extract CS from CFA by the inverted reflux classifier (IRC), covering single and multistage processing. The system consists of a series of parallel inclined channels mounted underneath an inverted liquid fluidized bed. This particular technique reported high recovery of CS, due to the so-called Boycott effect, which arises in inclined settling. The purity of the recovered CS was about 17% by volume, and recovery was 70%. However, by using all the three stages, the recovery fell to 50% and concluded that accelerated separation of the positively buoyant CS is promoted by the presence of a high concentration of the negatively buoyant CFA, and therefore, a single separation stage is preferable [21].

4.2. Dry Separation Method for Recovery of Cenospheres from CFA

The dry separation method is an alternative to the wet separation method, and has several advantages when recovering CS and other CFA particles [21]. For instance, in the dry-based method, particles are not subjected to liquid media, no chemicals (such as acetone) are involved, and there are no chemical reactions, and hence, no water pollution. Secondly, because there is no liquid media, the chemical composition of particles remains unchanged. Thirdly, since the particles are recovered in dry form, no drying process is required as in the case of the wet method. Thus, it is more economical because less electricity and space is needed [21].

For the separation of CS from CFA most widely used instrument is air classifiers. These types of instruments separate the particles are based on their geometry or aerodynamic equivalent diameter and density [63]. The separation of cenospheres from CFA by air classifiers relies on the airstream being applied in the air classifiers from the bottom. When this happens, the particles are subjected to gravity, and drag forces then in the direction opposite to the airstream applied. Thus, the heavier particles, whose terminal settling

velocity is more than the velocity of the applied air in an airstream, then, the particles will settle down against the airstream. While, on the other hand, in the case of lighter particles, whose terminal velocity is less than the applied air velocity, then the particles will move towards the air stream and finally will reach the top of the air classifiers [55].

Hirajima et al., 2010, recovered the CFA CS, and estimated the magnitude of separation for both wet and dry separation methods by applying the terminal velocity concept of particles. The diameter of the particles and density was used to calculate terminal velocity for separation efficiency of both dry and wet methods. The outcomes of this approach were confirmed by using a micron separator. The recovery of the cenospheres was only 4% lower than the estimated value (70%). By using a micro separator, recovery efficiency reached to 66%—which was almost equivalent to the estimated value of 70%. Furthermore, a group of investigators used micron separators to study the efficiency of the dry separation method for extracting CS from CFA [51].

Petrus et al., 2011, recovered CFA CS through employing a dry separation method that used closed-type pneumatic separator. The feed rate was maintained by keeping 20% CS in CFA. They also studied the particle size distribution of the recovered particles by using an LA-950 laser-scattering particle size distribution analyzer [57].

Wrona et al., 2020 [21], reported a novel dry separation method for the recovery of CS from CFA generated in Poland thermal power plants. The dry separation method is comprised of three sequential arrangements of techniques, in the following order: Firstly fluidized bed separation; secondly, screening; and finally, pneumatic separation. The first arrangement was associated with a mechanical activator to inhibit the agglomeration, and consequently, this step reduced the further processing of about 52–55% fractions of the cenospheric material by wt.%. Furthermore, the obtained fractions were individually transferred to another section, i.e., pneumatic separation. It was found that the CFA have initially 0.64 wt.% of CS of the total weight of CFA, i.e., CS concentrate that constituted about 17 wt.% of the initial fly ash. By applying this novel technique, the CS recovery efficiency reached up to 81%. Among all the raw materials present in CFA, almost 83% could be recovered by this dry novel separation method. The recovered CS could be further either purified or may be directly used in the various industries [21].

Some investigators have also used a triboelectric separation system to extract CS from CFA. Kiani et al., 2016, acomplished this by using an inverted reflux classifier (IRC), covering single and multistage processing. The system consisted of a series of parallel inclined channels mounted underneath an inverted liquid fluidized bed. This particular technique reported high recovery of CS, due to the so-called Boycott effect, which arises in inclined settling. The purity of the recovered CS was about 17% by volume, and recovery was 70%. However, by using all the three stages, the recovery fell to 50%. They concluded that accelerated separation of the positively buoyant CS is promoted by the presence of a high concentration of the negatively buoyant CFA, and therefore, a single separation stage is preferable [21,59].

5. Applications of Cenospheres in Ceramics and Environmental Cleanup

CS finds applications in all the fields which include building/construction materials [64], ceramics [65], plastics [28], construction [10], lightweight construction material [66], recreation [67], coating [31], automotive [28], paints and coatings [68], energy storage devices [69,70], polymer fillers [71], buoyancy and as low dielectric constant substrates [14]. They are best fitted for adding into silicone rubber to increase the conductivity. Besides this, it also increases rubbers' suitability as an electromagnetic wave absorbing material [71,72], which can be used in electronic, as a broad band microwave absorber, and radar-based applications [73].

5.1. Applications of Cenospheres in Environmental Cleanup

Due to its unique and remarkable features, it is widely used in environmental cleanup and building materials. Among environmental cleanup, it is used as a water purifying agent which helps to remove an excess of fluorides [74]. Until now, CFA have mainly been used for the adsorption of pollutants, such as dye, heavy metals, pesticides, etc., from wastewater. CFA as an adsorbent draws huge attention, due to their low cost, as mainly considered waste, have various structural materials like carbon, plerospheres, ferrospheres, and CS. Besides this, CFA have pores on their surface, which enhances the adsorption phenomenon. Thus, the adsorption is based on CFA is quite economical as the overall cost of remediation decreases drastically. There are few reports where, instead of whole CFA, their different structural particles have been used for wastewater treatment. For instance, CS are used after functionalization to remove specific organic or inorganic pollutants from wastewater [75]. However, the separation process of CS from CFA may increase the cost of the process, so it is less preferred. There are few instances where CFA and cenospheres have used as an adsorbent for air pollutant [22]. Moreover, there are several reports in the literature where cenospheres, extracted from CFA, were used for wastewater treatment. Markendya et al., 2014, utilized modified CS to remove disperse blue (DB) and disperse orange (DO) dye by batch adsorption study. The former removed 78%, while the latter removed 81% dyes from the 40 g/L concentrated aqueous solutions [50]. Markandeya et al., 2021, synthesized zeolites from CFA cenospheres and applied them for the remediation of disperse orange and disperse blue dyes from the textile mill effluent. The removal percentage of disperse orange (DO) was 96% under 119 min, while for disperse blue (DB), it was 95.23% under 122 min. The other parameters were almost similar for both the dyes. Finally, they concluded that the CS-derived zeolites are not only eco-friendly and economical; rather, they have the potential to remediate both DO and DB dye from the textile mill effluent [76].

Some investigators have also reported using these cenospheric particles after surface modification for the purification of wastewater. One investigation, led by Xu et al., 2011, showed the results of loading CS with magnesia, produced by a wet impregnation process with MgCl$_2$ [74]. CS can be used for the solidification and removal of liquid and other hazardous wastes, along with the production of porous crystalline molded glass [30].

5.2. Applications of Cenospheres in Construction

Several investigators have reported the applications of CFA CS in civil engineering and construction, due to their unique and remarkable properties [66]. As a construction material, these CFA CS are used as a refractory material [77] because of their low TC and high-temperature resistance as these are formed by aluminosilicates phases which are thermally stable [25].

5.2.1. Applications of Cenospheres for Concrete and Concrete with Lightweight Material

In the field of construction materials, CFA cenospheres display a significant reduction in concrete density, while retaining most of its mechanical strength [41]. Due to this property, CFA cenospheres have higher mechanical strength per unit weight of concrete, thus, CFA cenospheres are a suitable material for manufacturing lightweight concrete [78]. CS are micron-sized spherical particles, which act as mini ball-bearings in concrete mixture. Due to ball-bearing effect of CS [14], if added in the conventional concrete mixture, it will increase the workability by keeping CS dosage 1 and 5%. The ultra-fineness property of CS could also enrich 'finishability' and 'trowelability' of concrete [79]. CS have a property to act as structural aggregates, and thus, have a positive impact on the strength and density of the concrete that results in better packing [80]. Because CS have good water absorption capacity, they are added to concrete, allowing it to be desirably dry (water in concrete leads to corrosion) and also decrease its density [81,82].

CS can be used as an additive material for lightweight cement with reduced water release [83]. Souza et al., 2019, used the CS for manufacturing lightweight, high tensile strength concrete. For this investigation, they collected cenosphere from a Brazilian power plant, which has a total cenospheric content was 0.2% and size was varying from 30 to 300 μm. Concrete was manufactured by substituting 33, 67, and 100% FA by CFA extracted CS, which exhibited higher strength than 70 MPa, while the density was just 1500 kg/m^{-3}. It was found that the addition of CS as FA drastically improved the specific strength of high-strength concrete by keeping the equivalent mechanical properties [84]. CS are widely used to manufacture lightweight concrete which are used as slate, shale, or expanded clay. Cenospheres are used to replace FA because CS density (0.7 g/cm^3) is lesser than water (1 g/cm^3), thus preparing lightweight concrete. The size of cenospheric particles varies, as per effective concrete mix. Less cement is required to cover the particles when using the remaining cement as lubrication to increase the workability. Due to the incorporation of CS in concrete, there is improved TC and could be used as a noise barrier. Due to the incorporation of CS, the compressive strength of the cement reduces, but this can be overcome by using silica nanoparticles [85]. Likov et al., 1999, manufactured lightweight hydrated products from the CFA CS. They concluded that on the increase of hardening temperature, there is enhanced crystallization of the hydrated products due to the CS and expensive additives [79]. Hanif et al., 2017 developed an ultra-lightweight cementitious composite by utilizing using aerogel and CS. Due to the utilization of both materials, there was a significant reduction in the density of the composite materials [86].

5.2.2. Cenospheres as Replacement of Cement and Fine Aggregate (FA) from Concrete

The CS extracted from CFA have similar properties to that of CFA, i.e., cementitious and pozzolanic properties, which makes them a suitable material for the replacement of cement in concrete [80]. Other properties, such as bulk density, particle size, and surface area, help to accelerate the hydration process of cement. CS have been widely used as a partial substitute material of FA, due to the similarity in the structure [87]. Due to the replacement of FA, by CS six major properties of the concrete were affected, i.e., (1) Thermal conductivity; (2) workability; (3) bulk density; (4) surface finish; (5) mechanical strength; (6) porosity. Few investigators said that even after the addition of CS in concrete by replacing FA, there was no major loss in the mechanical strength of concrete. However, consequently, the concrete pores volume was increased. This may adversely affect the permeability of concrete. Satpathy et al., 2019, said that to achieve desired mechanical features, there should be an optimum percentage of both CS and sintered CFA, i.e., 50% and 75%, respectively [64]. Investigators replaced the FA with CS in 33, 67, and 100%, and found that there is a gradual decrease in the compressive strength of the concrete. Moreover, it was also found that the addition of CS in any form in the concrete to replace FA, resulting in the adverse effect of hydration; hence, CS should be added only 5 to 10% [64]. Table 1 is showing the percentage removal of CS in the concrete by various investigators.

Table 1. Percentage replacement by cenospheres in the concrete.

S. No	% Replacement	Density Kg/m^3	W/C Ratio	Reference
1.	10 20 30 0	1724 1592 1477 2200	0.32	[88]
2.	40 50 60 70	1506 1444 1342 1260	0.3	[80]
3.	10 20 30 5	1900 1700 1500	0.3	[86]
4.	50 75 100 30	1980 1867 1769 1612	-	[64]
5.	33 67 100 0	2134 1912 1650 1870	0.2	[84]
6.	50 75 100	- - -	-	[43]
7.	15 20			[89]

5.2.3. Cenospheres as an Insulating Material

CS extracted from CFA have a large amount of mullite, so the CS are used as mullite-coated diesel engine components [9]., steel soaking, heat exchangers, industrial furnaces for glass re-melting, refractories, and aluminum reclamation [90]. The incorporation of CS as an additive in concrete provides them extra sound and heat insulation, due to their hollow spherical shape. Moreover, cenospheres also help in the insulation (sound and thermal) properties of plasters/coatings [91], and mortars used for buildings [86]. Adding 40% cenosphere by volume to the cement matrix can increase the sound reduction coefficient by 100% [91]. The most recent applications of CS are in the field of energy storage, especially as a material for hydrogen storage [92]. Hanif et al., 2017, used aerogel and CFA extracted CS and developed a lightweight, composite material, which was also found to have excellent thermal insulation properties along with specific strength [86].

5.2.4. Application of Cenospheres in Ceramics

Qian et al. prepared porous mullite-based ceramics by gel casting method. This method procures mullite and extracts CS from the CFA [40]. The authors evaluated the impact of CS content and sintering on strength and density. The microstructure exhibited that CS could act as a sintering aid and a pore-forming agent. When the sintering temperature was high (1200 °C), then the obtained porous mullite ceramics was highly porous in nature, with low density and low thermal conductivity [40].

5.2.5. Application of Cenospheres as Fillers and Nanofillers

One of the major applications of cenospheres is as a cheap source of fillers. It is highly demanded in polymer and polymeric composite material, due to their high surface to volume ratio, which requires less binder, resin, and water to wet the surface. In the field of composites and nanocomposites, CS are used as lightweight fillers, due to their enhanced

properties and reduced cost of the final product [93]. As a filler, they are also used in grout, due to their light color and fine size, which makes them suitable candidates for applications in filling pinholes. CS are rich in Al and Si, which provide strength and hydration to binder agents. Packing of placed grouts has become much easier and effective, due to their perfectly spherical shape, scrubbing nature, and ability to roll in small holes. Danish et al., 2020, provided a detailed overview of the recovery and applications of CS extracted from CFA [14]. Krueger et al., 1966, utilized the CFA extracted CS for refractory purposes [77]. Some investigations concluded that using the CS as a nanofiller increased the porosity because of their spherical nature [94].

5.2.6. Cenospheres as an Internal Curing Agent

CS are porous in nature which is decided by the shell thickness, which is further governed by the alumina and silica content in the CFA. In order to become porous CS, there should be an optimum amount of alumina (directly) and silica (indirectly), which are associated with the shell's porosity and thickness [25]. CFA and CS have a high percentage of glass content, which could be eliminated by etching that will expose more pores on the surface of microspheres [95]. Due to this etching, the water holding capacity of the CS increases up to 180%, which releases the water very gradually from the cement mixture to reduce autogenously shrinkage of concrete [17]. This internal curing can increase the compressive strength of cement concrete/mortars [96]. Liu et al. also prepared foam concrete from the CFA CS [58].

One of the important draws back of the addition of CS to cement mortar for plaster is that it increases the porosity, which may increase the seepage. From the analysis, the author found that, initially, the strength of the cement paste increased after the addition of CS, but over time, strength decreased [80]. Due to the addition of CS, the increased pozzolanic activity plays an important role in the reduction of strength over the period. They have reported that 10% replacement of CS by weight is an optimum replacement of FAC, which resulted in the increase in the porosity, due to the addition of CS. The higher percentage of CS in the mix adversely affected the durability of the concrete. Wang et al., 2017, revealed the internal curing of high-performing concrete composed of CS. They created pores on the surface of CS by chemical etching, and found that the perforated CS water absorption capacity was 180%, and the loaded water could be easily released from CS under higher relative humidity (85%). Due to this internal curing, there was no autogenous shrinkage of the mortar; there was also high compressive strength of the cement mortar [97].

5.2.7. Application of Cenospheres in Manufacturing of Composite Material

As the CS are lightweight and high strength material, they are considered as a widely suitable material for the designing of modern lightweight composite materials that have high demand in the automotive and aerospace industry [98–100].

In the field of polymers, copper-coated CS and their incorporation in organic polymers, opens a new possibility to change the electric properties of polymers from an insulator to a conductive material [46]. CS can be coated or covered by various metals and act as a magnetic waves shield; therefore, it can be used in electronic and radar applications. Bora et al., 2016 and 2018, used the CS extracted from CFA, to prepare composite material, which was used as microwave absorber [73]; while, in another approach, polyaniline-cobalt coated the cenosphere composite film was used for electromagnetic interference shielding [101]. Nickel-coated CS display a potential for protection and microwave absorption applications [102]. Zeng et al., 2006, extracted the CFA CS, and then coated it with $BaFe_{12}O_{19}$ using a sol gel process. The $BaFe_{12}O_{19}$ was synthesized by using $Fe(NO_3)_3 \cdot 9H2O$, $Ba(NO_3)_2$, and citric acid as precursor materials. Furthermore, they thoroughly studied the structural properties of the developed material. They concluded that because of the even and continual Ba hexaferrite coating, the nonconductive CS acquired the magnetic properties [103]. Valdez et al., 2011, used the CFA extracted CS as an ultra-low-TC coating on the barriers. They used different temperatures to sinter CS. Low TC was observed with the nonsintered

one due to the presence of hollow air spaces in the CS, while sintering slightly boosted the heat conductivity due to the change in microstructures [49].

Irtiseva et al., 2021, developed a composite material, composed of recycled rubber, CS, and biobinder. Three such phase composites were prepared in two forms, i.e., block and granules, where the former was used to investigate mechanical features and density, while the latter form was used for absorption of oil and water products. The maximum compressive strength was when there was no CS and rubber was added, which was 79.3 MPa [71]. CS coated with nanosilica can be used to produce superhydrophobic surfaces [104]. The several composite materials that use CS very frequently are: Polyurethane composites, syntactic polymer foam, functionally gradient materials, high impact strength nylon composites, and polyester composites [105], as depicted in Figure 1. CS are widely used to manufacture several composite and nanocomposite materials that are favored by the cenosphere's particle size, spherical shape, inertness, etc. This investigation found that the sample contains a hollow structure with a magnetic property [106]. Tiwari and Bose, 2004, observed the effect that adding CS has on the acoustic properties of concrete. They said that the acoustic properties of concrete increased significantly with the reduction in the density of the concrete [91]. Cardoso and Shukla 2002, reported that due to the addition of CS in the concrete, its fracture toughness increased by 200% [107]. CS is being used as a filler in many polymer composites, due to its unique properties. Cenosphere can be coated with copper to manufacture conducting polymers to be used for electromagnetic shielding applications [108]. Some of the investigators have also reported that it is possible to manufacture LWHS material of polyester by controllable size and altered polyester [107]. By utilizing CS in polyethylene (high density), different colors could be obtained, which will simultaneously reduce the requirement of pigment [28].
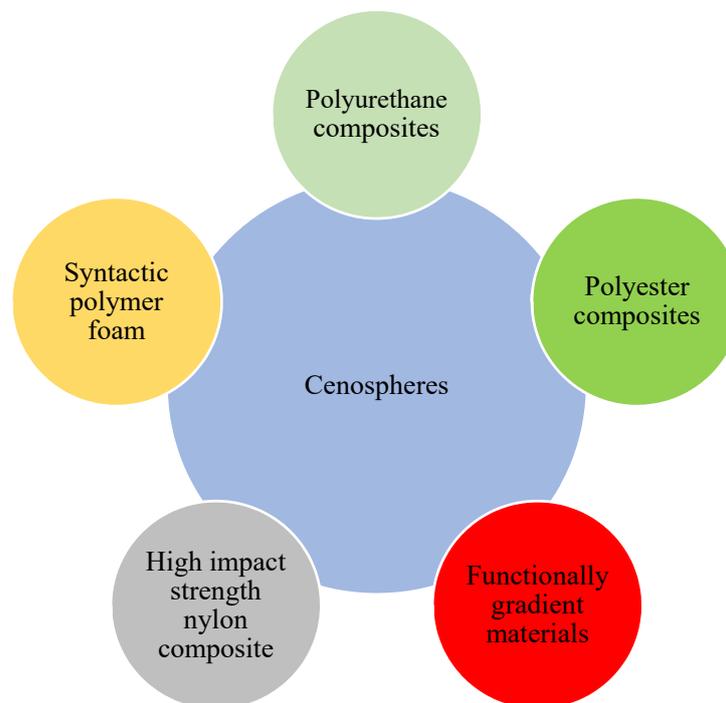

Figure 1. Applications of cenospheres as a filler in composites and nanocomposites.

5.3. Applications of Cenospheres in Syntactic Foams and Spheroplastics

Foams that are composed of CS are low weight-high strength [72], high resistance to gas erosion, can be cast in the desired shape, which make these foams desirable material in preparing high-performance products, such as heat insulators, thermal resistors, etc. [109]. CS-based ceramic insulation material was manufactured for reusable ceramic heat insulators for the space shuttle, which can protect the basic structure of the vehicle [30]. These

foams are manufactured by mixing matrix material and CS to obtain several beneficial properties like less moisture absorption, good buoyancy, high strength, high damage resistance, and energy absorption [110]. Due to all these unique properties of cenospheric foams, it is widely used in marine, aerospace, and automotive industries for developing materials that can take high hydrostatic loads [110].

One of the most emerging applications of CFA extracted CS in ceramics is their application as fillers in spheroplastics. Lisyatnikov et al., 2020, reported the production of spheroplastics with high dielectric current from the waste CFA on a solid fuel [111]. The only major problem is the presence of toxic heavy metals (Hg, Cu, Cr, Cd, Ni, As, Pb, Zn, etc.) along with the CS. Besides these heavy metals, some of the investigators have also reported the presence of few radioactive elements, which may have further negative impacts on the material, as well as the handlers.

5.4. Miscellaneous Applications of Cenospheres

CS are applied in several applications—for instance, phase change material, etc. Menenzes et al., 2016, studied the TC of CS, extracted from CFA, which was experimentally determined for particle diameter size ranges of <105 μm, 105 μm to 180 μm, and >180 μm using concentric spheres method [112]. Besides this, there are several reports where investigators have mentioned the applications of CFA CS paints and enamels. These CS acted as a reinforcement for the matrix of the polymer composites that can be added to the paints [113]. It has also been used in fluidized bed material [114], as reported by Berkowicz et al., 2016. Some of the researchers have used CS in the petroleum industry, for several purposes while digging wells. Moreover, some of them used CS as an additive in drilling mud, which not only helps in the drilling process but also increases the life span of the drilling equipment. It has also been used to prepare well cements that fill the space between the casting and well boundary—either to protect from groundwater or separate different wells. Well cements, prepared using CS, were not only lightweight, but also economical—which was bestowed with high strength and heat insulation properties [71]. Several investigators have also reported the application of CS in the pharmaceutical industry, as a carrier molecule for drugs, as well as in radiodiagonosis and cancer treatment [115,116] There is also the possibility of applications of modified CS airplane parts, defense components, due to their high mechanical strength. Table 2 shows the summarized applications of CS in various applications.

Table 2. Research that carried out in different fields incorporating cenospheres.

S. No.	Topic	Use/Study Outcomes	References
1.	Application in cementitious materials	Production of lightweight material. Improvisation of thermal and acoustic properties of cementitious materials.	[31]
2.	Effect on mechanical properties of cement-based composites	Reduced the shrinkage of cement-based composites.	[117]
3.	Alkali activated cenosphere as a binder for preparation of lightweight hemp-concrete	Provides compressive strength by using less energy. An alternative sustainable binder because of its lower carbon emissions without compromising on mechanical properties.	[118]
4.	Cenosphere for designing synthetic foams	Increases strength. Improves insulation capacity. Lightweight.	[119]
5.	Hydroxyapatite cenosphere for water treatment	Effective for treating heavy metals (lead and copper) contaminated water.	[120]

Table 2. *Cont.*

S. No.	Topic	Use/Study Outcomes	References
6.	CS for making long-lasting anode materials for batteries	Improves cycling stability. Increases rate capability and service life Improves safety	[121]
7.	CFA CS for the formation of lightweight concrete (LWC)	Increased cenosphere content decreases the density of concrete. Perfect for development of structural LWC.	[64]
8.	Cenosphere as fillers for flexural fatigue properties of thin cementitious materials	Improves strength and ductility and flexural fatigue	[122]
9.	Glass CS for development of lightweight cementitious composites	Outstanding in cementitious composites formation. Highly compatible with reinforced fiber matrix. Helps to improve the thermal insulation.	[123]
10.	Synthesis of nano-Ni coated CScomposites	Ease in processing. Lightweight. Cost-effective. Can be used in electromagnetic shielding.	[124]
11.	CS for lightweight ferrocement (green) formation	Increases compressive strength, ductility, and flexural strength. Improves durability.	[125]
12.	CS as filler in cement-based composites	Increases strength. Used in casting lightweight load-carrying structural members.	[126]
13.	Cenosphere's stability against alkali-silica reaction in cement composite	Protection from the alkali-silica reaction. Show pozzolanic reactivity, which increases with temperature.	[127]
14.	Formation of cenosphere concrete material	Increases water uptake due to the porous nature of CS. Improves equilibrium water content.	[17]
15.	CS as FA for development of lightweight high strength concrete	Increases compressive strength. Low porosity. Low density.	[84]
16.	Properties of asphalt concrete and cenosphere cement	Increment of 17% in density of concrete by replacing FA with CS. Increment of 40% in sound absorption by using cement with CS.	[91]
17.	CS for preparation of lightweight cementitious material	Improves mechanical properties. Reduces permeability. Increases durability. Improves thermal insulation.	[86]
18.	Cenosphere's use in development of lightweight concrete	Improves TC.	[128]
19.	Cenosphere's use for microencapsulating phase change materials (PCM) in concrete	Cost-effective. Increases thermal storage stability and capacity. Improves mechanical properties.	[129]
20.	CS for internal curing of high-performance concrete	Increases compressive strength. Removes of autogenous shrinkage. Reduces stress concentration.	[97]
21.	Cenosphere's effect on properties of CFA brick	Reduces in bulk density. Improves performance on cold region. Improves insulating properties. Financially sound.	[130]
22.	CS: Use in refractories	CS were used as feedstock to form innovative refractories. Cenosphere incorporation helped to meet enhanced performance characteristics.	[77]

6. Conclusions

CFA is considered hazardous waste, due to the presence of numerous heavy metals. However, it has been categorized into useable materials in the last decade, due to their minerals and structurally important structures, such as cenospheres, plerospheres, and ferrospheres. Cenospheres possess unique and remarkable physical, chemical, and mechanical properties; thus, it has drawn the attention of the scientist from all parts of the world in ceramics, environmental cleanup, and lightweight materials. Cenospheres could be either magnetic or nonmagnetic, based on the Fe content. The cenospheres could be recovered from CFA by both dry and wet-based methods, but the wet-based method is preferred because of their economical nature [59]. The efficient recovery of cenospheres was least affected by the morphological properties of the cenospheres. To date, cenospheres are successfully used in the field of ceramics, wastewater treatment, agriculture, and electronics, and soon they will be considered a material of the future.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

CS	Cenospheres
TC	Thermal conductivity
CFA	Coal fly ash
TPPs	Thermal power plants
LWC	Lightweight concrete

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