



Review article

A critical review of eggshell waste: An effective source of hydroxyapatite as photocatalyst

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Abstract

Interest in eggshell waste and its modification has been increasing rapidly. The outstanding property of eggshell waste is its high calcium carbonate content. This can be transformed to calcium oxide, and can provide a source of hydroxyapatite. As interest in clean technology has grown, the use of eggshell waste has become a focus of research. The objective of this review article is to characterize eggshell waste, describe the preparation of hydroxyapatite from eggshell waste and its modification, and discuss the use of hydroxyapatite as a photocatalyst.

1. Overview

As the global population continues to grow, engineering technology must also develop, in areas including infrastructure, automotive products, medical and pharmaceutical technology, and food packaging. However, even advanced technologies produce waste as a byproduct. These impact upon the water, air and soil environment. Governments have introduced a wide range of policies to address this. Such policies aim to minimize the use of hazardous chemical reagents and to control their toxicity [1,2]. Our research group has investigated the use of bacterial cellulose as a flexible substrate for electronic devices [3]. After modification, this can replace flat glass sheet substrates [4,5]. The goal of this project is to produce a model for waste management and utilization. We investigated the feasibility of adding value to a range of municipal wastes and bio-based agricultural products.

To the best of our knowledge, eggshell waste is considered to be municipal waste, and therefore imposes a disposal cost on the food industry. Under EU regulations, eggshell is considered to be a hazardous waste. It is discarded in hundreds of thousands of tonnes worldwide [6]. However, it can alternatively be viewed as a novel source of calcium [7,8]. Eggshell waste has a number of attractive properties, including high thermal stability, chemical resistance, and adsorption. It therefore has potential applications in sectors such as wastewater treatment and gas adsorption, and as a

heterogeneous catalyst and ion exchange membrane [9-13].

In chemical structure and composition, eggshell can be considered as a natural porous bioceramic. It comprises approximately 97% calcium carbonate [14]. This makes the production of bioceramic from eggshell challenging.

Eggshell waste has been proposed as a source of hydroxyapatite for photocatalytic uses, in which the rate of a photoreaction is increased. Photocatalysis has been used in advanced oxidation processes for water treatment to remove organic impurities. The principal use of photocatalysts has been in semiconductors such as TiO₂ [15,16], ZnO [17,18], Fe₂O₃ [19,20], and CdS [21,22]. We have successfully synthesized ZnO using conventional methods and investigated its photocatalytic properties in methylene blue degradation [23,24]. ZnO has potential applications as a photocatalyst in the decontamination of a wide range of organic dyes used in the textile industry. The mechanism of photocatalysis makes use of the radiation of energy to the band gap, generating high electron-hole pairs which increase the rate of reaction. Many strategies for increasing the efficiency of photocatalysts have been investigated, including doping with transition metal ions, surface coating, and surface sensitization [25-29].

Given the large volume of eggshell available from municipal waste, it constitutes a major potential calcium source. It can also provide a raw material for the preparation of hydroxyapatite. This

review article discusses the preparation of hydroxyapatite from eggshell and its potential use as a photocatalyst.

2. Synthesis and modification of hydroxyapatite from eggshell

2.1 Characterization, modification, and preparation of hydroxyapatite from eggshell

Eggshell is a major source of calcium. It constitutes approximately 10% of the complete egg, which is a basic global foodstuff consumed both in homes and industry. Large quantities of eggshell solid are therefore sent for disposal in landfills without pretreatment [30]. In structure, eggshell is a porous bioceramic, produced by sequential deposition of layers around the albumen in the hen oviduct. It has a perfectly ordered structure, with a polycrystalline organization throughout [31]. Eggshell comprises a foamy layer of cuticle, a calcite or calcium carbonate layer, and two shell membranes. More than 7000 funnel-shaped pore canals are distributed unevenly across the shell surface for water and gas exchange. Its composition is approximately 96% calcium carbonate, 1% magnesium carbonate, and 1% calcium phosphate and proteins. This makes eggshell waste a potential raw material for hydroxyapatite synthesis.

Hydroxyapatite is a useful material, with applications as a catalyst and ion exchange membrane, and in gas adsorption and medical settings. It is a phosphate with the formula $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, and has two alternative structures, being either monoclinic or hexagonal [32]. It has very low solubility at neutral pH, but dissolves slowly in an acidic environment.

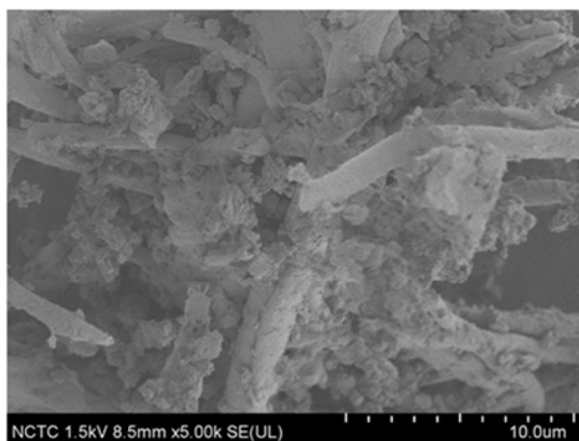


Figure 1. Morphology of eggshell waste [33].

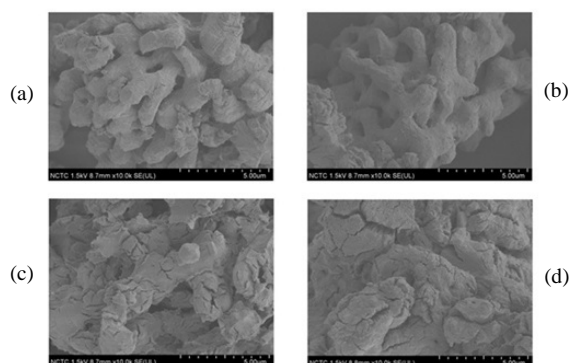
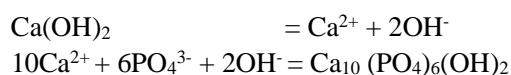


Figure 2. Morphology of calcium oxide produced by calcination of eggshell at (a) 700°C, (b) 800°C, (c) 900°C, and (d) 1000°C [33].

Our research group has focused on the eggshell research. Its high porosity makes it attractive for water and gas adsorption. When milled into a powder, it can be dispersed into a bacterial cellulose suspension, acting as a membrane [9]. Its other main use is as a calcium source for hydroxyapatite preparation. Laca et al. [6] note that eggshell is a naturally porous bioceramic. From the structural point of view, calcium carbonate can be transformed to calcium oxide at high temperatures. Ummartyotin et al. [33] investigated on the feasibility of using eggshell waste as a raw material for hydroxyapatite preparation. Figure 1 shows the typical morphological properties of eggshell waste. In structure, it is a crosslinked D fiber-mesh, with hierarchical features. The diameter of the fibers has been estimated to be between 10 nm and 1 μm in different orientations. As a calcium source, eggshell has been calcined at 700, 800, 900 and 1000°C for 2 h, producing calcium oxide. Figure 2 shows the morphology of the calcium oxide derived from eggshell waste, at a magnification of 10000x. The morphological properties are due to the irregular shape. The porosity is produced by CO_2 evaporation.

Hydroxyapatite is prepared using a coprecipitation technique. The eggshell is calcined, then used as a Ca source and an alkaline reagent in solution, with DI water as solvent.



The process is conducted at a low temperature for 5 h. The hydroxyapatite surface is also modified by chemical reaction.

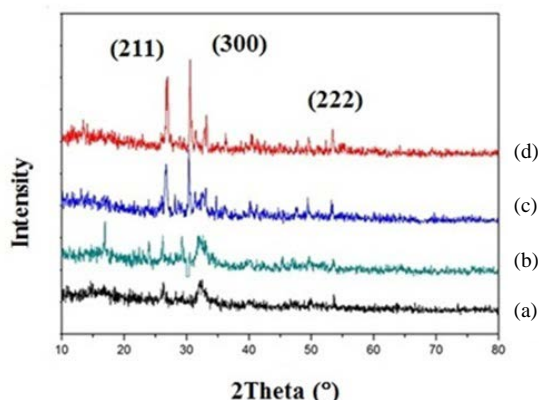


Figure 3. XRD pattern of hydroxyapatite prepared from calcium oxide at (a) 700°C, (b) 800°C, (c) 900°C, and (d) 1000°C [33].

Gergely et al. [34] observed the important role played by milling at the nano-scale. This has been performed using alumina balls and bowls [35]. The nano-scale eggshell powder is prepared using homogenous mixing, to prevent agglomeration of the calcined product. A second important factor is the heat treatment of the eggshell waste. This is done to avoid pH adjustment and control, utilization of organic solvent as pressure exposure. Wu et al. [36] discussed the role of heat treatment when preparing hydroxyapatite from crushed eggshell waste. Formation of hydroxyapatite is conducted at 1000°C for 1 h, to induce a reaction between the crushed eggshell and dicalcium phosphate dehydrate. The hydroxyapatite phase is similar to that in our XRD experiment, shown in Figure 3. Wu et al. [37] further proposed a hydrothermal process for hydroxyapatite synthesis. This has proven to be an effective and convenient method of batch preparation of hydroxyapatite, allowing control of the morphology and architecture [38]. The hydrothermal process is operated at 150°C for 24 h, and produces hydroxyapatite with a needle- or rod-like nanostructure. The as-synthesized hydroxyapatite

takes the form of grape peel, sweet potato peel, or pomelo peel.

As demand for hydroxyapatite has increased, a range of synthetic routes have been developed, producing hydroxyapatite with a variety of morphological properties. Sol-gel formation is one of the most effective techniques. Roopalakshmi et al. investigated the structural and morphological properties of hydroxyapatite prepared by this route [39]. The solvent used, aging time, and molar ratio of chemical reagent all help determine the size and shape of the hydroxyapatite produced. To control the size and morphological properties, cationic surfactants have been used to provide a template in hydrothermal processing. Shanthi et al. [40] investigated the role of cationic surfactants in nano-hydroxyapatite preparation. Their approach has a number of advantages. It can be run at ambient temperature and atmospheric pressure. The calcination process has been found to improve crystallinity. The peak positions are in good agreement with the JCPDS (09-0432), with lattice parameters $a = b = 0.9418$ nm and $c = 0.6884$ nm. Prabakaran et al. [41] reported the utilization of CTAB cationic surfactant as a template in hydroxyapatite preparation. The CTAB acts to regulate nucleation and crystal growth, and also plays a role in micelle concentration. The nano-scale hydroxyapatite was stable at temperatures up to 1100°C. Further research is needed on the reaction temperature, ageing time, and concentration when CTAB is used. Yang et al. [42] reported synergistic effects between citrate and CTAB. The ratio of citrate to CTAB affected the 3D hierarchical structure of the hydroxyapatite. Morphologically, hydroxyapatite can be prepared as hollow spheres, bunched microrods, or nanorod clusters. In previous work, our research group has investigated the morphological properties of clay modified by CTAB. The CTAB has been shown to increase the specific surface area and pore volume of the clay [43].

Table 1. Production of hydroxyapatite from eggshell waste and chemical reagent.

Technique	Raw material	Surface area	Morphology	Reference
Co-precipitation	Eggshell waste	75 m ² .g ⁻¹	Rod-like particles	Ummartyotin et al. [33]
Co-precipitation	Chemical reagent	45 m ² .g ⁻¹	Blocky particles	Shariffidin et al. [54]
Hydrothermal	Chemical reagent	125 m ² .g ⁻¹	Rod-like particles	Tanaka et al. [55]
Thermal extraction	Chemical reagent	20 m ² .g ⁻¹	Needle-like particles	Piccirillo et al. [56]
Co-precipitation	Chemical reagent	56 m ² .g ⁻¹	Blocky particles	Reddy et al. [57]

CTAB can also be used as a cationic surfactant in the preparation of europium (III)-doped hydroxyapatite. Kataoka et al. [44] demonstrated the production of Eu^{3+} doped hydroxyapatite nanocrystal in the presence of a cationic surfactant. The uniformity of shape and mono-dispersion of the nanocrystals were investigated. The Eu^{3+} was shown to be effectively positioned in the Ca sites in the hydroxyapatite structure, offering applications as a photoluminescent material. Duta et al. [45] produced titanium-modified hydroxyapatite using pulsed laser deposition. This increased the film roughness of the hydroxyapatite, suggesting possible uses in implant applications. Wang et al. [46] also demonstrated a self-assembly technique for production of silicon-modified hydroxyapatite. The properties of the modified hydroxyapatite make it a candidate for functional bone and tissue engineering. Hydroxyapatite can also be used as a host material for heavy metal ion substitution. Substitution by zinc, silver, and molybdenum allows hydroxyapatite to be used in medical technologies [47-49].

The choice of solvent is a key parameter in hydroxyapatite synthesis. From a fundamental point of view, the solubility of the calcium and phosphate sources in different organic solvents plays an important role in the phase equilibrium. Solubility data can be used to tailor the optimal crystallizer configuration and to select the appropriate solvent for the specific solute [50-51]. Chen et al. [52] investigated the thermodynamic parameters of the solid-liquid equilibrium in hydroxyapatite preparation. They investigated nine solvents: water, methanol, ethanol, 1-butanol, acetone, acetonitrile, ethyl acetate, dimethyl formamide, and tetrahydrofuran. The solubility of hydroxyapatite was shown to be a function of temperature for a pure solvent, and to be related to the crystallization process. In a binary mixture, the solubility of hydroxyapatite depends on the ratio of the solvents, and may be related to intermolecular bonding with the hydroxyapatite. Karimi et al. [53] demonstrated the synthesis of hydroxyapatite using deep eutectic solvent (DES). This is considered to be an environmentally friendly solvent. Ionic liquids are thought to form through hydrogen bonding with hydroxyapatite.

Table 1 summarizes the techniques and chemical reagents used in hydroxyapatite preparation, and the morphological properties of the product.

2.2 Preparation of hydroxyapatite from eggshell as composite

Although eggshell and hydroxyapatite offer many environmental benefits, their utilization is limited by their physical characteristics. When they are in powder form they cannot be used in applications involving vibrational force. To extend the use of eggshell and hydroxyapatite, composite materials have been developed. Figure 4 shows the morphological properties of an eggshell and bacterial cellulose composite. Eggshell particle was dispersed into a bacterial cellulose sheet, for use as a platform in water and vegetable oil adsorption.

Eggshell has also been proposed as a reinforcement agent in plastic. Li et al. [58] investigated the physical properties of poly(L-lactide) and functionalized eggshell powder composites. The addition of eggshell was reported to improve crystallization, the mechanical properties, and enzymatic hydrolysis. This bio-based composite has great potential for expanding the utilization of eggshell. Boronat et al. [59] successfully demonstrated an eggshell and polyethylene composite. Their goal was to use eggshell waste as a replacement for conventional calcium carbonate. The presence of eggshell in the polyethylene composite improved stiffness, hardness, and the flexural and tensile modulus.

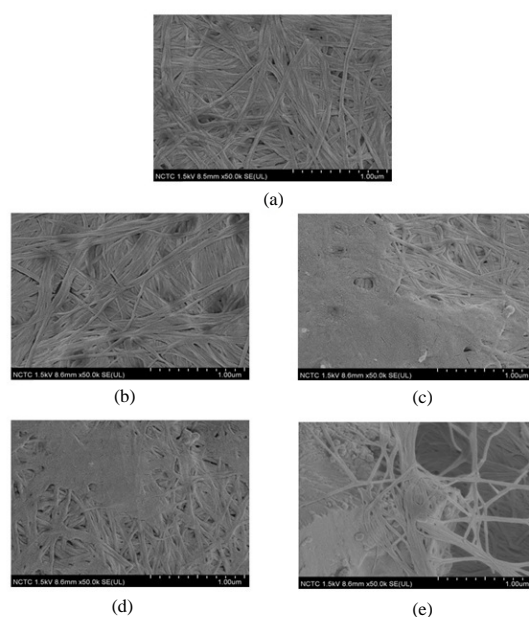


Figure 4. Morphological properties of eggshell and bacterial cellulose composite membrane (a) neat bacterial cellulose, (b) 1:0.5 ratio by weight of bacterial cellulose to eggshell, (c) 1:1 ratio, (d) 1:2 ratio, and (e) 1:5 ratio [9].

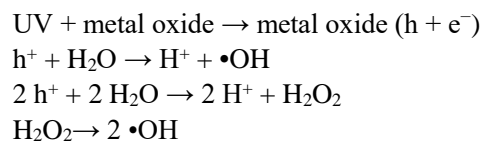
Both eggshell-based and hydroxyapatite-based composites have been successfully developed. However, utilization of hydroxyapatite has been limited by its physical characteristics. Key factors are its specific surface area and porosity. As noted in previous review articles [60-66], the main uses of hydroxyapatite have been in medical technologies. Mbarki et al. [67] investigated the effect of its high porosity. The mechanical properties of hydroxyapatite depend on the amount of pores, which is determined by the calcination temperature. Wang et al. [68] used computer simulations to investigate protein adsorption onto a hydroxyapatite surface by calcium or phosphate ions. These ions provide active sites for protein adsorption. Scudeller et al. [69] investigated insulin loading onto calcium phosphate as a drug delivery system for the oral treatment of diabetes and the stimulation of bone cell proliferation and bone mineralization.

Based on its porosity, hydroxyapatite has been developed as a hydrogel composite. Reaction with polymers such as alginate, chitosan, and cellulose forms hydrogel networks. Hydroxyapatite-based hydrogel composites can be used as adsorption media in heavy metal ion adsorption [70], as a scaffold material [71-74], and in catalysis [75-76].

3. Use of hydroxyapatite from eggshell as a photocatalyst

Photocatalysis is an advanced oxidation process that is used to degrade refractory organic and toxic pollutants present in water and treated sewage effluent into simple and harmless molecules. In chemistry, photocatalysis is used to accelerate a photoreaction in the presence of a catalyst. In this process, light is absorbed by a substrate. In photogenerated catalysis, the photocatalytic activity depends on the capacity of the catalyst to create electron-hole pairs, and to generate free radicals (e.g. hydroxyl radicals: $\bullet\text{OH}$), which undergo secondary reactions. Its practical application is made possible by water electrolysis using metal oxide semiconductors [77-81]. From the fundamental point of view, hydroxyapatite is a heterogeneous catalyst. It is used in a wide range of reactions: mild or total oxidation, dehydrogenation, hydrogen transfer, $^{18}\text{O}_2$ - $^{16}\text{O}_2$ and deuterium-alkane isotopic exchange, metal deposition, water detoxification, and gaseous pollutant removal. Its use as a heterogeneous catalyst involves the following mechanism.

Oxidative reactions due to the photocatalytic effect:



Reductive reaction due to the photocatalytic effect:

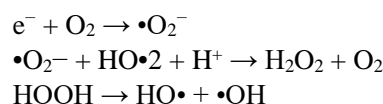


Figure 5 represents the photocatalysis reaction scheme for an inorganic semiconductor.

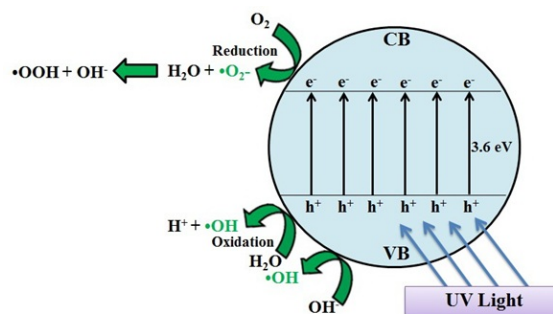


Figure 5. Photocatalysis reaction scheme for an inorganic semiconductor.

Piccirillo et al. [82] reported the use of calcium hydroxyapatite-based photocatalysts for environmental remediation. Hydroxyapatite photocatalysts can be prepared from unmodified hydroxyapatite, modified hydroxyapatite, doped hydroxyapatite, or multiphasic hydroxyapatite.

Numerous synthesis approaches have been used to vary the morphology and enhance photocatalytic efficiency. Liu et al. [83] successfully synthesized spherical nano-hydroxyapatite for use in methyl orange dye degradation at estimated sizes of 20-60 nm. They demonstrated 93% methyl orange degradation within 120 min under UV irradiation. Edralin et al. [84] used mussel shells as the raw material for preparation of nanorod shaped hydroxyapatite, and demonstrated 87% rhodamine B degradation after 300 min. Shariffuddin et al. [85], also using mussel shell, produced hydroxyapatite with high efficiency for azo dye degradation. For enhancing the photocatalytic efficiency of hydroxyapatite, structural modification is considered to be one of the most

effective methods. Partial substitution of transition metal ions in the calcium position has been used for energy band gap reduction. Liu et al. [86] investigated Fe(III) substituted hydroxyapatite. This catalyst is optimal for rhodamine B degradation. Gangarajula et al. [87] investigated strontium-substituted hydroxyapatite. The adsorption wavelength remained in the UV region at 320-340 nm, offering p-nitrophenol degradation. Feng et al. [88] successfully synthesized titanium-doped hydroxyapatite using a sol-gel technique. The specific surface area and porosity of the product allowed it to be used for photoelectrocatalytic reduction of hexavalent chromium.

Multiphasic hydroxyapatite has also been developed, and the role of hydroxyapatite and metal oxide semiconductor as a photocatalyst has been studied [89-90]. The development of hydroxyapatite and TiO₂ powder composite has also been investigated. Its high specific surface area and porosity allows it to adsorb gas, with the TiO₂ acting as photocatalyst. Xie et al. [91] developed a titanium and hydroxyapatite composite for photo-degradation of pentachlorophenol. Hydroxyapatite can adsorb molecules of the organic pollutant, which are then degraded by the TiO₂. Ono et al. used TiO₂ and hydroxyapatite for photo-oxidation of gaseous ethanol. The enhanced photocatalytic activity when compared with the TiO₂ powders was attributed to the deactivation of repeated injections of ethanol gas by the TiO₂ and hydroxyapatite composite. The development of hydroxyapatite and metal oxide photocatalysts has since been extended to include Fe₃O₄ and SiO₂ [92-93].

In chemical modification, Pickering emulsion is an important technique for modifying the specific surface area and porosity of hydroxyapatite. Pickering emulsion is a process in which solid particles adsorb onto the interface between two phases. If oil and water are mixed, small oil droplets form, and become dispersed throughout the water. As these droplets coalesce, they decrease the energy in the system. If solid particles are added, they bind to the surface of the interface and prevent the droplets from coalescing, stabilizing the emulsion [94-97]. Sun et al. [98] developed a hydroxyapatite-stabilized Pickering high internal phase emulsion for protein adsorption. The pore size was estimated to be in the range 20-50 μm. Adsorption was due to chemical reactions between the hydroxyl group and amino group. Zhang et al. [99] developed hydroxyapatite for the Pickering

emulsion technique. The ratio of water to oil was varied to investigate the pore size and specific surface area of hydroxyapatite.

4. Remaining challenges

Many studies have investigated the role of hydroxyapatite and its application as a photocatalyst, as interest in its industrial uses has grown. The use of eggshell waste as a raw material for hydroxyapatite has been described as a "Green Scenario" [2,100-102], given its abundance in municipal waste. The use of eggshell waste as a source of hydroxyapatite is a major contribution to environmental management. However, such uses remain limited. From the structural viewpoint eggshell is essentially calcium carbonate, and can be converted to calcium oxide and carbon dioxide at high temperatures. If eggshell waste is to be used as a raw material for hydroxyapatite preparation, its availability must be considered. Although eggshell is a major component of municipal waste, it varies greatly in quality [103,104]. When contaminants are present, mass production is challenging. Impurities become important when calcium oxide is used in co-precipitation with the phosphate group. The internal phase produced may affect electron and hole transfer during photocatalysis.

One important aspect is the dissociation of calcium oxide in an organic solvent. Calcium oxide is formed by covalent bonding between the calcium and oxygen atoms. These chemical bonds are very strong, making them difficult to dissociate at a low temperatures, increasing the energy needed for processing and negatively affecting the economics.

Reproducibility is key to mass production. Before commercialization is attempted, lab-scale testing should be used to iron out any problems in the mass production process. The variable quality of the eggshell will also impact on the reliability of processing. The quality control of eggshell waste is a major outstanding issue.

5. Conclusions and future outlook

Eggshell waste is major part of a municipal waste, and a potentially significant source of calcium oxide. It can also provide a raw material for hydroxyapatite preparation. Hydroxyapatite can be synthesized using calcium oxide and the phosphate group, and several techniques are available to transform it into a photocatalyst. To enhance its

photocatalytic efficiency, it has been prepared using transition metal substitution, as a biphasic with metal oxide semiconductor, and as a powder composite. Eggshell waste is a potential source of photocatalytic hydroxyapatite, offering new routes to environmental remediation and management.

6. Acknowledgements

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