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# The Preparation and Characterization of Different Types of Eggshells Acidified with Acetic Acid †

**Eliza-Gabriela Brettfeld [1,2], Daria-Gabriela Popa [1,3], Raluca Somoghi [1,4], Cristian Andi Nicolae [1], Adrian Birtas [5], Diana Constantinescu-Aruxandei [1] and Florin Oancea [1,3,*]**

1 Bioresources and Polymers Departments, National Institute for Research & Development in Chemistry and Petrochemistry—ICECHIM, Splaiul Independenței Nr. 202, 060021 Bucharest, Romania; eliza.brettfeld@icechim.ro (E.-G.B.); daria.popa@icechim.ro (D.-G.P.); raluca.somoghi@icechim.ro (R.S.); cristian.nicolae@icechim.ro (C.A.N.); diana.constantinescu@icechim.ro (D.C.-A.)

2 Faculty of Chemical Engineering and Biotechnology, National University of Science and Technology Politehnica Bucharest, Splaiul Independenței Nr. 313, 060042 Bucharest, Romania

3 Faculty of Biotechnologies, University of Agronomic Sciences and Veterinary Medicine of Bucharest, Bd. Mărăști Nr. 59, 011464 Bucharest, Romania, Romania

4 Faculty of Petroleum Technology and Petrochemistry, Petroleum-Gas University, B-dul Bucureşti, Nr. 39, 100680 Ploiești, Romania

5 Renault Technologie Roumanie, Str. Preciziei, Nr. 3g, 062202 Bucharest, Romania; adrian.birtas@renault.com

* Correspondence: florin.oancea@icechim.ro; Tel.: +40-213-16307

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**Abstract:** This paper investigates the acidification of eggshells of different origins with acetic acid. The acidification process was investigated for conventional and organic eggshells generated from the production of liquid eggs in the food industry and hatched eggshells from egg incubators. The acidified eggshell materials were characterized using Fourier-transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM) analysis, and thermogravimetric analysis (TGA). The results demonstrate that each type of investigated eggshell generates different nanostructures due to slight variations in their composition and this indicates potential applications: as a source of calcium supplements or to produce a snow-melting agent or $CO_2$ adsorbent.

**Keywords:** liquid eggs; hatched eggs; side-streams; nanostructures; organic content



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## 1. Introduction

Eggshells are a major side stream of the agro-food industries, generated during liquid egg production [1] and from the hatchery process [2]. Eggshells have been considered for applications in various fields, such as calcium supplements for food and feeds [3], snow-melting agents [4], soil conditioners/biofertilizers [5], as a potential toxic element sequestrant [6,7], for the generation of porous CaO used as a catalyst in trans-esterification reactions [8], or for $CO_2$ absorption [9]. These practical utilizations of eggshells correspond to the circular bioeconomy approach, aiming to extract the maximum value from agro-industrial side streams, promoting their reuse, recycling, and repurposing [10–12].

Despite being a natural byproduct of avian egg production and reutilization, the improper disposal and accumulation of eggshells can have detrimental effects on the environment [1]. There are various aspects to this issue, including the impact on landfills (occupying valuable landfill space and having a long lifespan), water bodies (affecting aquatic ecosystems and compromising water quality), and global warming (the release of methane, a potent greenhouse gas). Additionally, the release of harmful substances during decomposition and the potential for leaching hazardous compounds into the soil are concerning due to the possibility of disrupting soil ecosystems and altering soil chemistry.

Understanding the environmental risks associated with eggshell waste is crucial for developing effective waste management strategies and promoting sustainable alternatives [13]. This includes methods such as their utilization for $CO_2$ capture within the circular economy framework. In line with this concept, eggshells are a sustainable resource for $CO_2$ capture [9].

The excessive emission of $CO_2$, a major greenhouse gas, is a significant contributor to global climate change [14]. Developing efficient and cost-effective $CO_2$ capture methods is crucial for mitigating these adverse effects.

Chemical looping is a promising technology for efficient and cost-effective $CO_2$ capture [15–19]. This approach utilizes solid carriers, such as calcium oxide (CaO), to cyclically capture and release $CO_2$ through redox reactions [20]. The reversible reaction between CaO and $CO_2$ enables the separation of $CO_2$ from flue gases without the need for energy-intensive separation processes. In recent years, significant research [21,22] efforts have been dedicated to understanding the fundamental mechanisms and optimizing the performance of CaO-based chemical looping systems [23] for $CO_2$ capture.

Acetic acid applied as pre-treatment to eggshells generates a mesoporous structure and improves their $CO_2$ capture efficiency [24]. Formic and citric acid applied to eggshells were also proven to promote the formation of a more porous CaO structure with increased $CO_2$ uptake capacity [25]. The acidulation process with acetic acid also improves the fluidization behavior of the porous CaO resulting from eggshells [26].

The calcium acetate resulting from the acidification of the eggshells with acetic acid has other applications, such as calcium supplements [27,28] or snow-melting agents [4].

This work explores the potential applications of materials from the acidification of different eggshells: conventional and organic eggshells from the food industry/liquid eggs, and hatched eggs from eggs incubators. The results suggest that ecological eggshells are more suitable for producing food supplements, and hatched eggshells are more suitable for use as a $CO_2$ adsorbent.

## 2. Materials and Methods

### *2.1. Materials*

Three types of eggshells, conventional and organic eggshells from the food industry/liquid eggs and hatched eggs from an egg incubator, were used. The conventional (commercial) eggshells were obtained from Avicola (Lumina, Romania). The organic eggshells were supplied by Cortina Bioprod (Curtisoara, Romania). The hatched eggs were obtained from Hipocrate (Bucharest, Romania). Acetic acid (Merck Group, Darmstadt, Germany) was utilized as an organic acid. Calcium oxide and calcium carbonate, p.a. (Sigma-Aldrich, Merck Group) were used as reference material for FTIR analysis.

### *2.2. Eggshells Treatment with Acetic Acid*

The reactions of eggshells with acetic acid can be described as follows:

$$CaCO_3 + 2CH_3COOH = Ca(CH_3COO)_2 + H_2O + CO_2 \quad (1)$$

The eggshells' treatment with acetic acid was performed according to Nawar et al. [25,29]. The eggshells were thoroughly washed with water to eliminate impurities such as egg white remnants, straw, flakes, and blood traces. Following the washing process, the cleaned eggshells were allowed to dry overnight. This step facilitated the complete evaporation of residual moisture. The dried eggshells were subjected to grinding using a centrifugal mill (S 100, Retsch, Verder Group, Haan, Germany) equipped with stainless steel balls within the grinding chamber. For acidification, acetic acid was utilized as a 1M solution. Six grams of eggshells were carefully added to 60 mL of the respective acid solution, taking precautions to manage the formation of foam. Gentle agitation was applied until the foam dispersed. Subsequently, the sample was heated at 90 °C in a laboratory oven (UE200 Memmert, Buechenbach, Germany) and stirred at 500 rpm on a magnetic plate (Arex 6, Usmate Velate, Italy), for 2 h. Following the acidification process, the treated sample was left to dry overnight at 105 °C, ensuring complete evaporation and the drying of any remaining moisture. The acidified and dried eggshells underwent another grinding step to obtain homogenous samples.

### *2.3. Characterization Techniques*

The acidified eggshells were characterized using Fourier-transform infrared spectroscopy (FTIR) and transmission electron microscopy (Tecnai G2 F20 TWIN Cryo-TEM, FEI Company, Hillsboro, OR, USA). TEM analysis allows for examining the microstructure and morphology of the acidified eggshells, providing valuable information on their physical characteristics.

The TEM system utilized a 200 kV scanning/transmission electron microscope (S/TEM) equipped with a TWIN lens and a high-brightness field emission electron gun (FEG). The sample for TEM examination was prepared by pouring a droplet of the aqueous dispersion on a formvar copper grid without staining.

FTIR analysis was used to identify chemical functional groups and their modification after acidification. The Attenuated Total Reflectance (ATR) mode was used, with a wavelength range of 7800–400 $cm^{-1}$, a peak resolution of 4 $cm^{-1}$, and 45 scans per sample. The analysis was performed using an IR-TRACER-100 FTIR (Shimadzu, Kyoto, Japan). The spectra were recorded in transmittance mode (%T).

The thermogravimetric analysis was performed using a thermobalance (Q5000IR, TA Instruments, New Castle, DE, USA) under the following conditions: high-temperature platinum (HT Pt) crucible of 100 µL, heating rate of 10 °C/min up to 1000 °C, initial sample mass 10 mg, purge gas 1: Nitrogen (99.999%) at 50 mL/min.

## 3. Results and Discussions

### *3.1. TEM*

The three types of eggshells (conventional, organic, and hatched) were studied in both their untreated and acidulated forms using organic acid—acetic acid (AA)—Figure 1.

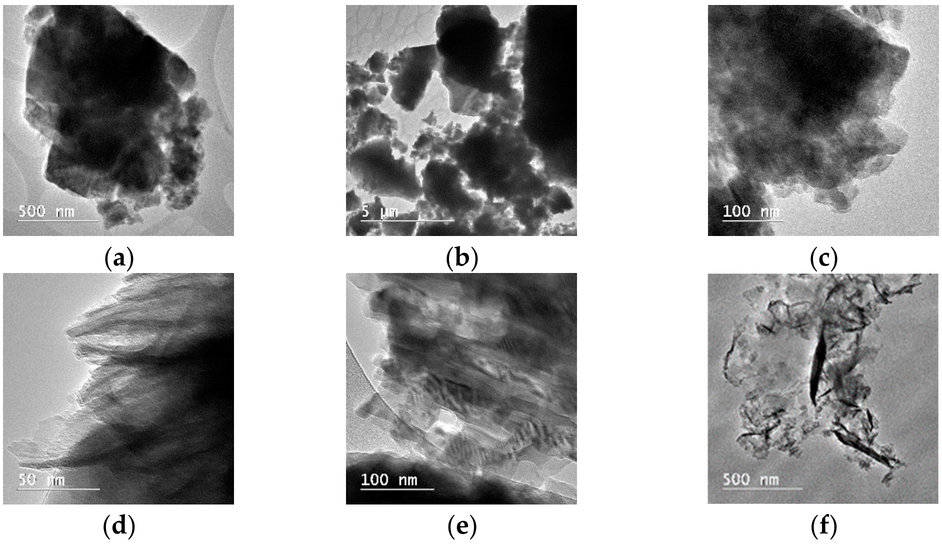


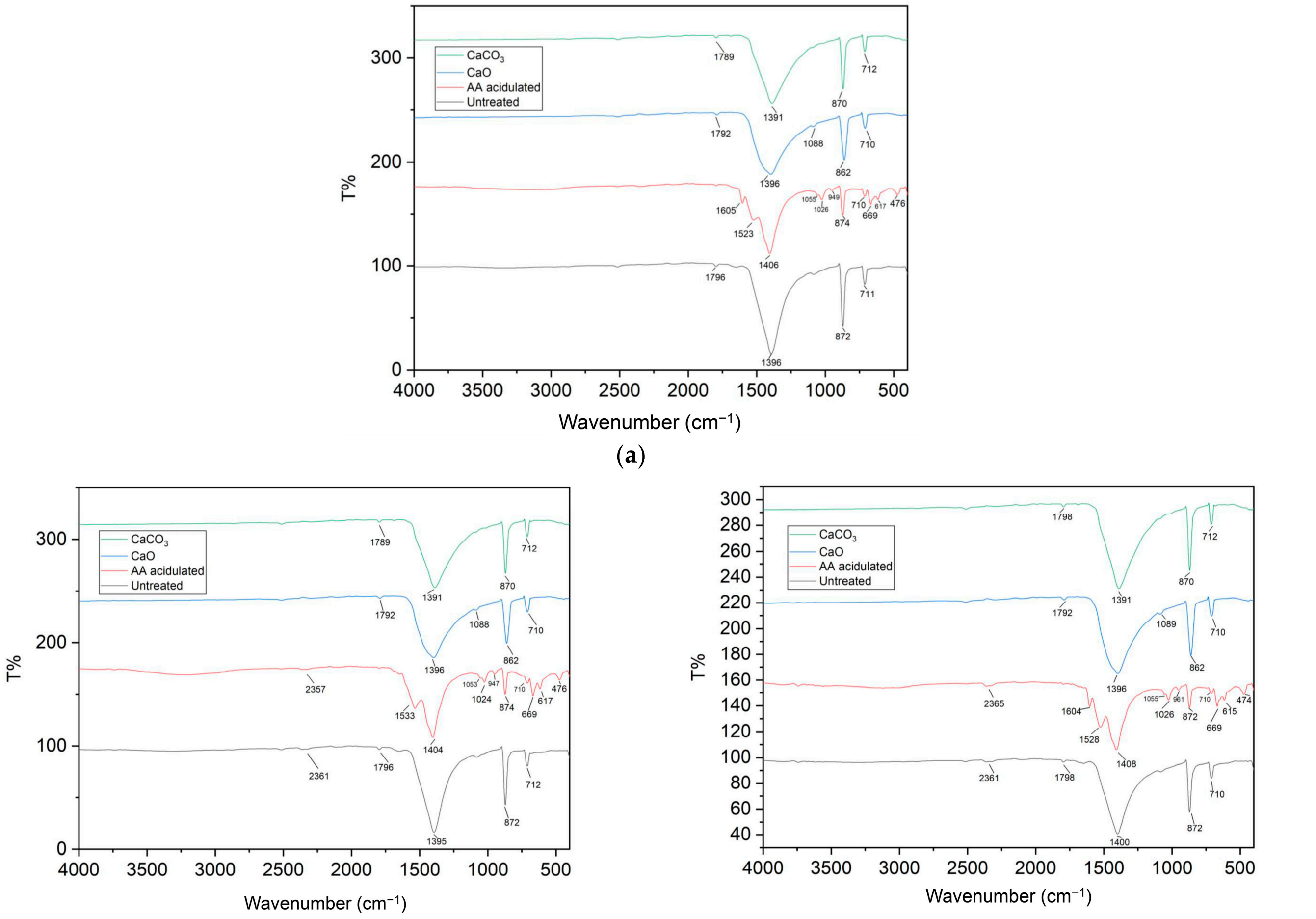

**Figure 1.** Transmission electron microscopy images of the acetic acid acidified eggshells. (**a**) Untreated conventional eggshells; (**b**) untreated organic eggshells; (**c**) untreated hatched eggshells; (**d**) conventional eggshells treated with acetic acid; (**e**) organic eggshells treated with acetic acid; (**f**) hatched eggshells treated with acetic acid.

In the case of untreated eggshells (Figure 1a–c), irregular-shaped (micro)particles ranging in size from 20 nm to 0.5–1 µm were observed, with no significant differences. After acidification, each type of eggshell exhibited a different morphology. Conventional eggshells treated with acetic acid appeared filamentous and homogeneous (Figure 1d). Organic eggshells acidified with acetic acid retained a rectangular shape (Figure 1d), in contrast with the eggshells from the conventional-growth hens. Acidulated hatched eggshells showed a distinct acicular shape (Figure 1e).

TEM analysis also revealed the porous nature of the treated and untreated crystallites [30,31]. It was reported that the nucleation and growth of CaO crystals from $CaCO_3$ resulted in the acidulated particles having a rougher surface than the untreated ones [25,26].

### 3.2. FT-IR

By comparing the spectra of the acidified and untreated samples with pure $CaCO_3$ and CaO (Merck) (Figure 2), as well as with the FTIR database, it was found that the untreated samples contained over 90% $CaCO_3$, evident from the vibrational bands of C-O in the carbonate molecule, ranging between 1394 and 1406 $cm^{-1}$, with a lower intensity for untreated eggshells.




(**a**) (**b**) (**c**)

**Figure 2.** The FTIR spectra of the acidified and untreated samples compared with pure $CaCO_3$ and CaO. (**a**) Conventional eggshells; (**b**) organic eggshells; (**c**) hatched eggshells.

Using the FTIR technique, differences between the untreated and acidulated eggshells are noticeable. No significant differences were observed among the three types of eggshells (conventional, organic, and hatched), even when compared to the pure $CaCO_3$ and CaO substances. Asymmetric stretching and an in-plane deformation [30] of C-O bonds were observed at wavelengths of 871 $cm^{-1}$ for untreated eggshells and 873 $cm^{-1}$ for eggshells acidulated with acetic acid. The C-O bond is also present in the wavelength range of 1570–1604 $cm^{-1}$ from carbonate. The band observed at 1777–1795 $cm^{-1}$ is attributed to the C=O bond in carbonate, which is only present in untreated eggshells regardless of their origin. The absence of this peak in acidulated eggshells suggests that the associated reactions occurred.

The Ca-O bond is expected to be around 500 $cm^{-1}$ and 710–712 $cm^{-1}$ [13]. These peaks are associated with the characteristic vibrations of calcium oxide (CaO) [32], but are not prominently visible in the recorded spectra. A noticeable difference in all acidulated eggshells is observed in the 440–670 $cm^{-1}$ region, where several detectable peaks are present, unlike in the untreated eggshells with no signals. This region can be associated with inorganic Ca-O bonds. Additionally, the range of 781–1055 $cm^{-1}$ suggests the absence of $CaCO_3$, the main component of the eggshell, as it has been converted to CaO after acidification and drying. These peaks are unique to the acidulated sample, suggesting the presence of organic acid residues (acetic acid) and possible changes in the crystalline structure of the eggshell material due to the acid treatment. Acetic acid has characteristic bands at 1176, 1780, 1795, and 3581 $cm^{-1}$ [33], which were absent in the acidified and calcined samples. This finding suggests that the eggshells' reaction and the calcination step entirely consumed the acetic acid. The peaks at 2359 $cm^{-1}$ are attributed to traces of amines and amides from the protein content of the shell and/or residual membrane remnants. Residual water traces (O-H bonds) are weakly present in the 3200–3400 $cm^{-1}$ range, indicating hygroscopic adsorption and the formation of $Ca(OH)_2$.

### 3.3. Eggshells' Thermal Decomposition

The results obtained from the thermal decomposition testing of acetic acid-treated eggshells, containing calcium acetate, measured at a constant rate of 10 °C/min up to 1000 °C, are consistent with literature data. Figure 3 and Table 1 highlight the mass loss percentage over three temperature intervals.

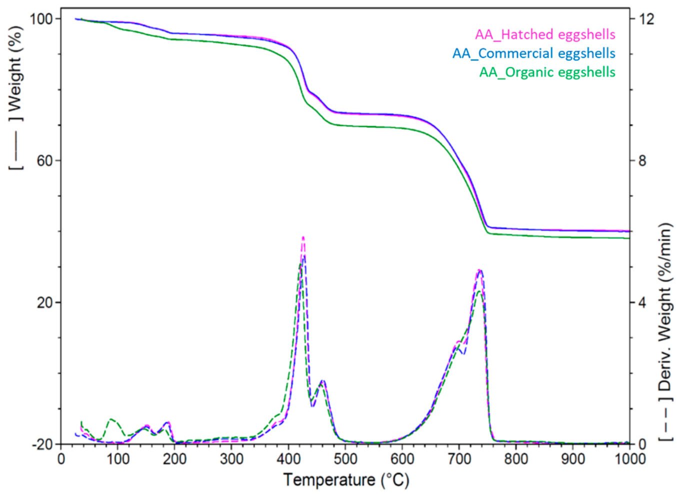



**Figure 3.** The decomposition of calcium acetate derived from various assortments of eggshells, conventional/commercial, organic, and hatched.

**Table 1.** TGA results regarding the decomposition of calcium acetate derived from eggshells.

| Eggshell Sample | 40–205 °C | 205–550 °C | | 550–850 °C | | 850–1000 °C | Residue | |
|---|---|---|---|---|---|---|---|---|
| | Wt. loss | Wt. loss | Tmax | Wt. loss | Tmax | Wt. loss | 850 °C | 1000 °C |
| | % | % | °C | % | °C | % | % | % |
| Hatched | 4.23 | 22.83 | 426.2 | 32.48 | 735.0 | 0.21 | 40.44 | 40.23 |
| Conventional | 4.16 | 22.68 | 427.3 | 32.78 | 737.8 | 0.46 | 40.38 | 39.91 |
| Organic | 5.89 | 24.59 | 421.2 | 30.95 | 735.3 | 0.49 | 38.55 | 38.07 |

An initial mass loss between 40 and 205 °C is attributed to the removal of water molecules and traces of acetic acid. In the range of 340–550 °C, the dehydrated calcium acetate decomposes to form acetone and $CaCO_3$:

$$Ca(CH_3COO)_2 \rightarrow CaCO_3 + C_3H_6O \quad (2)$$

Reaction (2) is observed as a combined percentage mass loss of 23–25%, with the reaction reaching maximum velocity at 421–427 °C. Calcium carbonate then decomposes to form calcium oxide between 600 and 765 °C, with the reaction reaching its peak rate at 735–738 °C.

In the evaluation of the thermal decomposition characteristics, a detailed comparison of the weight loss profiles of the different eggshell types emerges, shedding light on their distinct behaviors across various temperature ranges:

- Weight loss, 40–205 °C: Organic eggshells exhibited the highest residue weight (5.89%), while commercial eggshells had the lowest (4.16%). Hatched eggshells had a residue weight of 4.23%.
- Weight loss, 205–550 °C: ecological eggshells also had the highest weight loss (24.59%) at a maximum temperature of 421 °C, with commercial (22.68%) and hatched (22.83%) eggshells following closely, but at higher temperatures, 426–427 °C.
- Weight loss, 550–850 °C: commercial eggshells had the highest weight loss (32.78%) in this range, at 738 °C, whereas hatched (32.48%) and ecological (30.95%) eggshells had slightly lower values, but also at a lower temperature, namely 735 °C.
- Weight loss, 850–1000 °C: ecological eggshells exhibited the highest residue weight loss (0.49%), followed by commercial (0.46%) and hatched (0.21%) eggshells.
- Commercial eggshells had the highest weight loss (40.38%) at 1000 °C, followed by hatched (40.44%) and ecological (38.55%) eggshells.
- Residue weight loss: ecological eggshells had the lowest residue weight loss (38.07%) over 1000 °C, while hatched (40.23%) and commercial (39.91%) eggshells had slightly higher values.

Upon analyzing the weight loss profiles, it becomes evident that of the different eggshell types, ecological eggshells generally exhibit a slightly faster rate of decomposition due to their higher organic matter content, while commercial and hatched eggshells tend to decompose at slightly slower rates.

The thermal analysis results, as presented in Table 1, underscore the consistent behavior of calcium acetate decomposition across diverse eggshell assortments. The observed mass loss percentages and temperature intervals align with the established literature [25,26,29], affirming the reliability of the conducted experiments and shedding light on the successive stages of decomposition.

## 4. Conclusions

Our investigations demonstrate that each type of the considered eggshells generates different nanostructures due to slight variations in composition. Organic eggshell, with a higher organic content, maintained its rectangular shape, and was less efficient in forming nanoporous CaO. The acidification, with acetic acid, of conventional eggshells generates structures that are suitable for use as a snow-melting agent or as a $CO_2$ adsorbent. The hatched eggshell, most probably due to its lower protein content, generates an acicular shape, more efficient in producing nanoporous CaO, and more efficient as a $CO_2$ adsorbent.

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