Synthesis of second phase hybrid ceramics using two different bio-source and a comparative study on their morphological characterization

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Abstract. Ceramics are generally synthesized with various sources and methods. The most common method for synthesis of ceramics with reduced cost and energy is SOL-GEL method. Combustion synthesis is also a most widely used method for ceramic synthesis. In general, ceramics have enhanced hardness and dimensional stability even at elevated temperatures. For this reason, they are used in the production of refractories, thermal barrier coatings, chemical resistant coatings, wear resistant coatings, and also as reinforcement material to produce metal matrix composites and polymer matrix composites. This work concentrates on the comparison of morphological characterization of such reinforcement particles synthesized from different sources. The particles size range varying from 7 μm to 250 μm with flaky and spongy structures are observed in the ash of Vicia faba. However, the ash of Cocos nucifera resulted in fibrous structure with a diameter of 50 μm to length above 600 μm, particles size ranging from 10 μm to 70 μm, micro tubes of diameter 3.6 μm to length of 150 μm. The EDX and XRD analysis of Vicia faba showed the presence of carbon as the major element with a few other elements.

1. Introduction

Ceramics are synthesized as glasses, poly crystals, single crystals, and in many forms dictated by their end use, including fine powders, monoliths, thin films, and composites. It paved way for several inventions like ultrahigh-strain single crystal piezo electrics, new high frequency dielectrics, phosphors intercalation compounds, ductile layered structural ceramics, and thermo electrics.

Ceramics are generally classified as traditional ceramics and synthetic ceramics. Thermal conductivity is low for ceramics in most of the cases, although there can be some ceramics with a temperature dependent conductivity and may go into superconductivity range at elevated temperatures. In this work, seeds of Vicia faba and leaves of Cocos nucifera is taken as the sources for synthesis of ceramics. This paper concentrates more on morphological study of synthesized particles and compare the obtained results.
2. Experimental Work

2.1. Materials and Pre-Treatment

In this work, seeds of a plant named Vicia faba are used. The seeds collected from the farmer after through checking for quality. The leaves of Cocos nucifera were also collected from the same farm which is actually a waste. Pre-treatment is given to the seeds and leaves by washing them in running water to remove the unwanted contaminants from them. Followed by washing, seeds alone are boiled in water and milk and it is not done for leaves. This is because the seeds in general, contains organic compounds with very low boiling point and could vaporize even at a low temperature. Seeds of Vicia faba are bought from farms directly for combustion synthesis. Pretreatment of seeds are made to avoid unwanted evolution of gases during combustion. Seeds are collected from the farms and are dried under sun for two days. After drying, seeds are washed in running water and dried under sun shade. After washing, seeds are boiled in water for one hour. Then again it is boiled in milk (if milk is not available dissolve 25G of milk powder in 100ml of water) for one hour. Followed by drying under the sun. After drying these seeds, they are checked manually for moisture. If moisture is still present the seeds are roasted to remove the moisture content. After roasting the seed is ready for burning.

Initially 500 g of seed is taken. After pre-treatment and drying, the weight of seed is found to be 419.392 grams. This indicates that the removal of excess moisture and some organic impurities from the seed has taken place. In one part of the pre-treatment, the seed is taken as it is without powdering and pre-treated. During burning excessive evolution of gases are found giving unpleasant smells. In another section, powdered seed is taken for pre-treatment.

The organic compounds are generally classified into water soluble and fat soluble. So that, while boiling in water and milk, the organic compounds are also removed accordingly. Whereas the leaves do not contain such compounds and the impurities present in leaves generally vaporize on burning. The leaves were roasted at a temperature of 300°C for 2 hr to remove the surface moisture and unwanted volatile compounds.

2.2. Burning of Leaves and Seeds:

The seeds after drying under the sun are weighed before burning. After weighing the required amount of seed, they are filled in the crucible and burnt in muffle furnace at 750°C for 3 hr to produce the ash. Then the crucible with ash is removed from the furnace and cooled in air. After sometime, ash samples are collected and stored. Ash particles may get attached to each other due to a weak mechanical bonding. This bonding is broken by simply subjecting them to manual crushing. Leaves are burnet at (i) 800°C for 2 hr and (ii) 950°C for 2 hr. Once burning is done, ash from each condition are separated and allowed for cooling.

3. Results and Discussion

3.1. Visual observations

The time taken for drying and burning is reduced and also evolution of gases is highly minimized or suppressed when the seed is used in powdered condition. Also when we use powdered seed we can burn comparatively high quantity of seeds. Hence efficiency is increased to some extent by using powdered seeds. When the seed was burned at 500°C, blackish product is obtained which contains some unburned seeds. This is due to the presence of volatile impurities that were unreacted during burning process. This unburned seed is found at the bottom of the crucible. It implies that sufficient amount of air is not available at the bottom layer for burning. Hence again the seed is burned at 750°C for 3 hours in the
muffle furnace with intermediate manual agitation by doing this the bottom layer comes to the top and the top comes to the bottom, Which results in uniform burning of seeds. While burning at 750° C a mixture of greyish and black powder is obtained which is taken for characterization. While burning leaves of Cocos nucifera, no such evolution of gas is observed and also the leaves burned completely and formed blackish grey ash. The obtained ash is observed like micro tubes and small particles. They are further subjected to sieve analysis to study the size distribution of the particles. Since the greyish colour ash was completely burned, it is taken for characterization.

3.2. Sieve analysis

The sieve analysis of burned seed infers that the more amount of fine powders are obtained when the seeds are burned after crushing instead of burning as it is. Also it the reduction in burning time is observed in powdered seed. On sieving the burned ash of Cocos leaf, it is observed that when the burning temperature increases the size of the particle is reducing. This is observed because of removal of volatile impurities at elevated temperature.

3.3. SEM Analysis

The SEM images of the burned seed with varying size is represented in the figures 1, 2, 3, and 4, particles ranging from coarser to very fine. The maximum particle size of 210 micron meter and minimum of 7 micron meter is observed. Coarse particles formed spongy structure (cauliflower structure), size
ranging from 102 μm to 210 μm with high amount of porosity which is generally not preferred because ceramics are naturally hard and hence it has apparent density and tap density more or less equal. So compact ability of ceramics is difficult, so it is difficult to compact ceramics. When porosity is present in high amount the compact ability of the ceramics will further reduce and high energy is required for compaction. If it is used for reinforcement, this porosity may be an initiation size for crack growth and also it accelerates the crack propagation thereby affecting the properties of the composite.

Intermediate particles formed flaky structure (fern like structure) with size ranging from 40-90 μm. This particles has less porosity than coarser particles. The compact ability of the intermediate particles are preferably good because they have a considerably difference between apparent density and tap density. But they have sharp corners, this sharp corners may act as initiation of tri axial stress in a reinforced composite. The development of tri axial stress is not preferred as it failures the material much earlier of its working lifetime. So to blend this corners, ball milling is preferred before reinforcement which will also results in uniformly distributed particles with respect to size and shape of the particle.
Micro particles formed flaky structure (rose petal like structure) with a size ranging from 15-40 μm with moderate amount of porosity. In this microstructure needle like structure is observed size ranging from 20-30 micron meter. This elongated structure has good compact ability and moderate advantage in reinforcement.

In very fine particles, flaky structure (cluster of leaves like structure) with particle size ranging from 7-20 μm is observed. In this structure blunt flakes are observed instead of sharp corner edges. The porosity is also comparatively low. The difference between apparent and tap density is relatively high and hence it has good compact ability. These particle are also preferred for reinforcement because of the presence of blunt corners instead of sharp. But the uniformity in distribution of particles with such morphology is low. Hence it is preferred to undergo milling to attain uniform particle size and shape distribution. Few particles are cylindrical in structure, few are acicular, and few are rounded structure.

The SEM images of the burned seed with varying size is represented in the figures 5, 6, 7, 8, 9, 10, and 11. Where figures 5, 6, and 7 represents the SEM image of leaves burned at 800 deg Celsius and figure 8, 9, 10 and 11 are the SEM images of leaves burned at 950 deg Celsius.

![Figure 5: Fine particles](image1)

![Figure 6: Intermediate particles](image2)

![Figure 7: Coarse particles](image3)

In this, particles of size ranging from 10 μm to 100 μm is observed. The particles are rod like and flaky structured. Few dendrites are also observed. The intermediate particles are observed under SEM. The morphology of the particles shown rod like structure with diameter of 50 μm to length of 600 μm. Few particles of 50 to 200 μm are also observed. The final particles remained in the sieve is taken and analysed under SEM. The formation of tubes of diameter 100 μm and length above 1000 μm is observed.
Particles of size ranging from 10 μm to 50 μm are observed. The particles formed are cylindrical in nature with blunt ends. Few particles are formed like thin needles with diameter of 5 μm. In figure 9, particles of size ranging from 50 μm to 100 μm are observed. The morphology of the particles are rod like structure. The whiskers of diameter ranging from 20 μm to 50 μm is observed in figure 10. The whiskers are fractured at some part which may be occurred during handling of the particles. Particles of size ranging 300 μm are seen in figure 11. Upon magnifying further, fine tubes formation is observed with diameter of 1 to 5 μm.

3.4. EDS Analysis

Table 1. EDX analysis of burned powders in % mass are shown below.

|     | % C | % O | % P | % Mg | % Fe | % K  |
|-----|-----|-----|-----|------|------|------|
| S1  | 65.69 | 8.55 | 3.63 | 0.65 | 5.38 | 16.47 |
| S2  | 81.23 | 4.62 | 1.99 | 0.18 | 3.21 | 8.77  |

EDX analysis confirmed the presence of carbon as the major element in the obtained ash. Other than carbon, few other elements are present in a considerable amount.
3.5. Methanol Test

To verify the presence of carbon in organic form, the ash is dipped in methanol solution. The ash precipitated down indicating the presence of organic carbon in both ash of seed and leaves as shown above. Generally organic carbon are more reactive than inorganic carbon. Also the bond nature of organic compound make it highly active at elevated temperature.

4. Conclusions

The ash of both source is characterized and compared. The ash of the seed resulted in particles of flaky structure. New structures like rosette structure, cluster leaves structure and cauliflower like structures are observed. The composition analysis of the burned ash infers the presence of carbon as the major element. The particles of size ranging from 7 μm to 250 μm are observed.

The ash of burned leaf shows various particles morphology like whiskers, micro tubes, rods, fine tubes and bars. The particle size ranging from 10 μm to 250 μm is observed. Tubes of 100 μm diameter and 1000 μm length is observed. Few particles are observed with dimensions of 500 μm, but they are composed of fine tubes of diameter 1 to 5 μm.

Methanol test verifies the presence of carbon is organic in nature. In addition to carbon, other elements like phosphorous, potassium, magnesium, copper, iron are also observed.

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