Original Article

Bean pod ash nanoparticles a promising reinforcement for aluminium matrix biocomposites

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1. Introduction

Metal matrix composite (MMCs) materials are being used in a wide range of structural applications in the aerospace, construction and automotive industries due to their lightweight and high specific stiffness and strength [1]. One sector where the use of composite materials is still evolving is the automotive industry. Composite materials offer great potential in reducing vehicle weight, thus increasing fuel efficiency and reducing CO₂ emissions. In addition to weight reduction, the number of individual parts can be significantly reduced making the high-volume composite car concept cost effective [2].

In its most basic form a composite material is one which is composed of at least two elements working together to produce material properties that are different to the properties of those elements on their own. In practice, most composites consist of a bulk material (the ‘matrix’), and reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix. This reinforcement is usually in fibre/particles form [3].

The high cost of current MMCs, interfacial reaction and high density of most common ceramic reinforcement compared to aluminium alloys has inhibited production on a large industrial scale, for example in the automotive industry. In attempts to over come this limitation, several research and development (R&D) programmes [4–6] were focused on the reinforcement of aluminium-based MMCs using low cost biomaterials from plant origin as reinforcement. This could reduce the cost and the weight of energy intensive metals for potential applications in engineering components for a new generation of vehicles.

The use of natural filler for the reinforcement of the composites has received increasing attention both by the academic sector and the industry. Natural filler have many significant
advantages over synthetic filler and fibres such as their light-weight, low cost, ability to reduce abrasion of machinery and also non-toxicity. Currently, many types of natural fillers have been investigated to be used in the industry including flax, hemp, wood, wheat, barley, and oats [7]. They are now fast evolving as potential alternatives to inorganic or synthetic materials for various applications as building materials and automotive components. The ever-increasing demand for low cost reinforcement stimulated the interest towards production and utilization of using bye-products from plant origin as reinforcement since they are readily available or are naturally renewable at affordable cost.

1.1. Biocomposites

Composite materials comprising one or more phase(s) derived from a biological origin are called biocomposites. In terms of the reinforcement, this could include plant fibres such as cotton, flax, hemp and the like, or fibres from recycled wood or waste paper, or even by-products from food crops. Regenerated cellulose fibres (viscose/rayon) are also included in this definition, since ultimately they too come from a renewable resource, as are natural ‘nano fibrils’ of cellulose and chitin [8].

There are several biomass resources such as bagasse from sugarcane, bark and wood waste, palm waste, corn waste and rice husk. All of this biomass waste can be modified and been used as other useful products.

Advantages of natural fibers over traditional reinforcing fibers such as glass and carbon are: low cost, low density, acceptable specific properties, ease of separation, enhanced energy recovery and biodegradability. Even these bio-composites maintain a balance between economics and environment allowing them to be considered for applications in the automotive, building, furniture and packaging industries [8].

1.2. Bean pod

Bean is a common name for large plant seeds used for human food or animal feed of several genera of the family Fabaceae (alternately Leguminosae). The term bean originally referred to the seed of the broad or fava bean, but was later expanded to include members of the New World genus phaseolus, such as the common bean and the runner bean, and the related genus vigna [9]. The term is now applied generally too many other related plants such as old world soybeans, peas, chickpeas (garbazos), vetches and lupins.

Bean is sometimes used as a synonym of pulse, an edible legume, though the term pulses is usually reserved for leguminous crops harvested for their dry grain. The term bean usually excludes crops used mainly for oil extraction (such as soybeans and peanuts), as well as those used exclusively for sowing purposes (such as clover and alfalfa). Leguminous corps harvested green for food, such as snap peas, snow peas, and so on, are not considered beans, and are classified as vegetable crops.

Beans pod are a waste by product of agricultural processing of beans seed. Across the globe, much research efforts in recent times are geared towards possible ways of recycling wastes for reuse to keep the environment clean and safe [6]. Bean plant (Parkia Biglobosa), is the material resource required for the production of beans pod. The harvested fruits are ripped open while the yellowish pulp and seeds are removed from the pods. The empty pods are the needed raw material. The pods make up 39% of the weight of the fruits while the mealy yellowish pulp and seeds make up 61%. The pod ash is used for soap making and for dying the traditional indigo clothes [9].

2. Metal matrix composite

Metal matrix composite materials have found applications in many areas of daily life for quite some time. Often it is not realized that the application makes use of composite materials. These materials are produced in-situ from the conventional production and processing of metals. In traffic engineering, especially in the automotive industry, MMCs have been used commercially in fiber reinforced pistons and aluminium crank cases with strengthened cylinder surfaces as well as particle-strengthened brake disks [10].

These innovative materials open up unlimited possibilities for modern material science and development; the characteristics of MMCs can be designed into the material, custom-made, dependent on the application. From this potential, metal matrix composites fulfil all the desired conceptions of the designer. This material group becomes interesting for use as constructional and functional material, if the property profile of conventional materials either does not reach the increased standards of specific demands, or is the solution of the problem. However, the technology of MMCs is in competition with other modern material technologies, for example powder metallurgy. The advantages of the composite material are only realized when there is a reasonable cost performance relationship in the component production. The use of a composite material is obligatory if a special property profile can be achieved by application of these materials [11].

The possibility of combining various material systems (metal-ceramic-non-metal) gives the opportunity for unlimited variation. The properties of these new materials are basically determined by the properties of their single components. The reinforcement of metals can have many different objectives. The reinforcement of light metals open up the possibility of application of these materials in areas where weight reduction has first priority. The precondition here is the improvement of the component properties. The development objectives for light metal composite materials are [12]:

- Increase in yield strength and tensile strength at room temperature and above while maintaining the ductility or rather toughness.
- Increase in creep resistance at higher temperatures compared to that of conventional alloys.
- Increase in fatigue strength, especially at higher temperatures.
- Improvement of thermal shock resistance.
- Improvement of corrosion resistance.
- Increase in Young’s modulus
- Reduction of thermal elongation
2.1 Composition of metal matrix composites

MMCs are made by dispersing a reinforcing material into a metal matrix. The reinforcement surface can be coated to prevent a chemical reaction with the matrix. For example, carbon fibers are commonly used in aluminum matrix to synthesize composites showing low density and high strength. However, carbon reacts with aluminum to generate a brittle and water-soluble compound Al₄C₃ on the surface of the fiber. To prevent this reaction, the carbon fibers are coated with nickel or titanium boride [13].

2.1.1. Matrix

The matrix is the monolithic material into which the reinforcement is embedded, and is completely continuous. This means that there is a path through the matrix to any point in the material, unlike two material sandwiched together. In structural application, the matrix is usually a lighter metal such as aluminum, magnesium, or titanium, and provides a compliant support for the reinforcement. In high temperature application, cobalt and cobalt-nickel alloy matrices are common [14].

2.1.2. Matrix alloy systems

The selection of suitable matrix alloy is mainly determined by the intended application of the composite material. With the development of light metal composite materials that are mostly easy to process, conventional light metal alloys are applied as matrix materials. In the area of powder metallurgy special alloys can be applied due to the advantage of fast solidification during the powder production. Those systems are free from segregation problems that arise in conventional solidification. Also the application of systems with oversaturated or metastable structures is possible [7].

2.2 Reinforcement

The reinforcement material is embedded into the matrix. The reinforcement does not always serve a purely structural task (reinforcing the compound), but is also used to change physical properties such as wear resistance, friction coefficient, or thermal conductivity. The reinforcement can be either continuous, or discontinuous. Discontinuous MMCs can be isotropic, and can be worked with standard metalworking techniques, such as extrusion, forging or rolling. In addition, they may be machined using conventional techniques, but commonly would need the use of polycrystalline diamond tooling (PCD) [15].

Continuous reinforcement uses monofilament wires or fibers such as carbon fiber or silicon carbide. Because the fibers are embedded into the matrix in a certain direction, the result is an anisotropic structure in which the alignment of the material affects its strength. One of the first MMCs used boron filament as reinforcement. Discontinuous reinforcement uses "whiskers", short fibers, or particles. The most common reinforcing materials in this category are alumina and silicon carbide [16].

Reinforcements for metal matrix composites have a manifold demand profile, which is determined by production and processing and by the matrix system of the composite material. The following demands are generally applicable [17].

I Low density.
II Mechanical compatibility (a thermal expansion coefficient which is low but adapted to the matrix).
III Chemical compatibility.
IV Thermal stability.
V High Young’s modulus.
VI High compression and tensile strength.
VII Good process ability.
VIII Economic efficiency.

These demands can be achieved only by using non-metal inorganic reinforcement components. For metal reinforcement ceramic particle or, rather, fibers or carbon fibers are often used. Due to the high density and affinity to reaction with the matrix alloy the use of metallic fiber usually fails. Which components are finally used, depends on the selected matrix and on the demand profile of the intended application.

The production, processing and type of application of various reinforcements depend on the production technique for the composite materials. Every reinforcement has a typical profile, which is significant for the effect within the composite material and the resulting profile. The group of discontinuous reinforced metals offers the best conditions for reaching development targets; the applied production technologies and reinforcement components, like short fibers, particle and whiskers, are cost effective and the production of units in large item numbers is possible. The relatively high isotropy of the properties in comparison to the long-fiber continuous reinforced light metals and the possibility of processing of composites by forming and cutting production engineering are further advantage [18].

2.3 Mechanism of reinforcement

The characteristics of metal matrix composite materials are determined by their microstructure and internal interfaces, which are affected by their production and thermal mechanical prehistory. The microstructure covers the structure of the matrix and the reinforced phase. The chemical composition, grain and/or sub-grain size, texture, precipitation behaviour and lattice defects are of importance to the matrix. The second phase is characterized by its volume percentage, its kind, size, distribution and orientation. Local varying internal tension due to the different thermal expansion behaviour of the two phases is an additional influencing factor [19].

With knowledge of the characteristics of the components, the volume percentages, the distribution and orientation, it might be possible to estimate the characteristics of metallic composite materials. The approximations usually proceed from ideal conditions, i.e. optimal boundary surface formation, ideal distribution (very small number of contacts of the reinforcements among themselves) and no influence of the component on the matrix (comparable structures and precipitation behaviour).
3. Production of the composites

This section describes the details of processing of the composites and the experimental procedures followed for their characterization and mechanical evaluation. The beans pod used in this work was obtained from ‘Ekpoma town in Edo state Nigeria (see Fig. 2). The equipment for this research are metal mould, sieves, digital weighing balance, hack saw grinding machine, hydraulic press, compounding machine, molding machine and bending and tensile testing machine, scanning electron microscope (SEM), X-ray diffractometer (XRD). X-ray fluorescent XRF, DTA/TGA machine, pin on disc wear machine.

The bean pods were cleaned to remove the dirt and were dried for about 20 h. The cleaned dried bean pod was calcined at 850 °C for 5 h. The calcined bean pod was used in the production of the 55 nm nanoparticles using sol-gel method. The composites were produced by double stir casting and double layer feeding method using aluminium alloy of type A2009 (3.7%Cu and 1.4%Mg) and 1–4 wt% of bean pod nano particles was added. Details method of the production of the bean pod ash nano-particles and composites are described elsewhere by the author and his co-workers [6,9].

X-ray diffraction analyses were carried out using a X’Pert Pro model diffractometer to identify the phases present, The microstructures of as-cast nano-composite were examined by a JEOL J100 SEM [6]. The hardness values of the samples were determined (ASTM E18-79) using the Rockwell hardness tester on “B” scale (Frank Welltest Rockwell Hardness Tester, model 38506) with 1.56 mm steel ball indenter, minor load of 10 kg, and major load of 100 kg and hardness value of 101.2HRB as the standard block.

The tensile properties of the as-cast composites sample were conducted on Tunis–Olsen tensile testing machine with a strain rate of $2 \times 10^{-3}$ S$^{-1}$. The test pieces were machined to the standard shape and dimensions as specified by the American Society for testing and Materials. The impact test of the as cast composites sample was conducted using a fully instrumented Avery Denison test machine. Charpy impact tests were conducted on notched samples. Standard square impact test sample measuring $75 \times 10 \times 10$ mm with notch depth of 2 mm and a notch tip radius of 0.02 mm at angle of 45° was used.

The Instron versatile fatigue testing machine of 100 KN was used for testing. Fatigue tests were carried out according to ASTM 3479 [14] and the stress ratios, $S_{\text{max}} / S_{\text{min}}$ was $-1$, where $S_{\text{max}}$ and $S_{\text{min}}$ are the maximum and the minimum applied stresses respectively and a frequency of 10 Hz were applied.

The samples for the wear test were polished with 1000 grit SiC emery paper and cleaned with acetone. The wear test was conducted using high temperature pin on disc wear testing machine. The standard disc used for testing was made of

Fig. 1 – Photograph of some natural fiber [8].

Fig. 2 – Photograph of Bean pod(Bio-Reinforcement).
hard steel ASE 1055 with hardness of 269 BHN whose surface roughness was 0.1 μm.

3.1. Results and discussion

Fig. 1 showed the XRD patterns of bean pod ash nanoparticle sample obtained by sol-gel method. The particle size of the samples has been calculated employing the Scherrer equation [6]:

\[ D = \frac{K\lambda}{\beta \cos \theta} \]  (1)

Where \( \theta \) is the angle between the incident and diffracted beams (degree), \( \beta \) the full with half maximum (rad.), \( D \) the particle size of the sample (nm) and \( \lambda \) the is wavelength of the X-ray. The results of XRD confirmed the formation SiO\(_2\), NaAlSi\(_2\)O\(_6\), CaCO\(_3\) and Al\(_4\)O\(_4\)C of nanostructure (see Fig. 3). It can be clearly observed that the diffraction peaks appear in the pattern corresponding to phase with good crystalline nature. The grain size of the prepared bean pod ash nanoparticles is found to be about 55 nm.

The structural morphology of the nanoparticles was investigated using SEM. Fig. 3b, showed the SEM image of bean

![Fig. 3](image)

**Fig. 3** – (a) The XRD pattern of bean pod ash nanoparticles. (b) SEM image of bean pod ash nanoparticles at 5000 and 40,000 magnification.
pod ash nanoparticles. The spherical shaped particles with clumped distributions are visible through the SEM analysis. From the SEM it is observed that the BPA nanoparticles are roundish with some angular in shape and small amount of particles are longitudinal in shape.

The SEM micrograph of the aluminium alloy composite reinforced with approximately 0 and 4 wt.% BPA nanoparticle are shown in Fig. 4a and b. The micrograph shows uniform distribution of the reinforcement particles, in either case, no pores have been observed indicating better wettability between the matrix and reinforcement particles. The microstructure of the unreinforced Al–Cu–Mg alloy is shown in Fig. 4a. The structure reveals the eutectic phase containing Cu₃Al₂, Al₆CuMg₄ in α-aluminum matrix. In the Al–Cu–Mg alloy, Cu and Mg are present in solid solution as Cu₃Al₂ and Al₆CuMg₄ phase both in the grain and along the grain boundaries. Fig. 4b showed the microstructure of the reinforced alloy with BPA nanoparticles additions. The microstructure reveals that there are reasonably uniform distributions of BPA nanoparticles particles in the metal matrix. The ceramic phase is shown as white phase, while the metal phase is dark. These structures are in agreement with phases studied by other researchers [16].

The results of the hardness values are shown in Fig. 5a. It is observed that hardness of bean pod ash nano-particles (BPA) reinforced composite is more than that of unreinforced alloy. It can be attributed to the higher hardness of ceramic particles compared to aluminum matrix alloy. The hardness of composite depends on the hardness of the reinforcement and the matrix. This is because of the number and the total surface area of BPA nano particles increased with increasing BPA nano particles weight percent. An average hardness value
of 46.7 HRB and 67.3 HRB were obtained for the 0 and 4 wt% BPA nanoparticle reinforce composites, respectively.

The results of the tensile strength are shown in Fig. 5b. Increasing the weight percent of reinforcing particles the tensile strength improved. The increases in tensile strength of the MMNCs is due to dislocation generation and accumulation and assuming the dislocations to be uniformly dispersed in the metal matrix. The residual dislocations are likely to be trapped at the reinforcing BPA.

The results of the impact energy are shown in Fig. 5c. From Fig. 5c, it was observed that there is no much decreases in the values of impact energy, e.g. the impact energy from 7.81 to 7.68 J at 0 and 4 wt% BPA nanoparticles respectively.

The fatigue testing was performed for constructing the S-N curve. An S-N curve for the AMCs with different particle size of reinforcement is shown in Fig. 5d. The results indicated that the fatigue strength of the composite was higher than that of the unreinforced alloy for any given number of cycles. The improvement in fatigue life is more pronounced when the stress level become lower.

Fig. 6a showed the wear rate of the studied materials as a function of the sliding distance. Two main features can be seen: the wear rate increases as the sliding distance increases from 100 to 1000 m, also the wear rate decreased as the wt% BPA nano-particles increases from 1 to 4 wt% in the composites. During the experiment interaction between the matrix and the counterpart of the disc was controlled by the BPA nanoparticles, this type of interaction is known as adhesion that exists between soft matrix and counter-face. In increasing the BPA nanoparticles phase, the amount soft matrix that are in contact with the hard counterface phase decreased this was attributed to increased in wear resistance of the composites when the weight fraction of the BPA nanoparticles are raise from 1 to 4%.

The friction co-efficient are shown in Fig. 6b. From the Fig. 6b, it was observed that the friction coefficient showed three phase evolution for each wt% BPA nano-particles: the first friction coefficient varied between 0.13 and 0.19, second varied 0.2–0.3 and then increases up to 0.45 at the three stages. A small difference is detected in the first step and a large difference is observed specially at the end of the second and the third steps. The variation of $\mu$ is essentially depending on the hardness values of the various composites. It was observed that the higher the hardness values the raise in the friction coefficient.

The Toyota Carina one (I) connecting rod was produced with composites of Al-3.7%Cu-1.4%Mg/4 wt% bean pod ash nanoparticles. The selection of this grade of composites was based on the best properties. The properties of this grade of composite are showed in Table 1.

The auto-card design of the connecting rod is showed in Fig. 7a. After removal the casting, the photograph of the connecting rod is showed in Fig. 7b. The low and idle speed engine system was used for the performance test, the test was con-
ducted according to recommended standard and similar work [14]. The engine used was a Toyota Carina model 1 of 12 valve. The engine is 1.5L single overhead camshaft version. It has a four stroke, four cylinder compression ignition engine type.

From the performance test analysis, it was observed that the fuel consumption for both of connecting rods are similar (see Fig. 8) for example the fuel consumption of the standard connecting rod varied from 0.430 to 0.761 kw/h (see Fig. 8a) and that of the developed connecting rod varied from 0.350 to 0.650 kw/h (see Fig. 8b). From Fig. 8b, it was observed that the fuel consumption of the developed connecting rod is lower as the duration of the running of the engine increases.

![Fig. 6 - (a) Variation of wear rate with sliding distance. (b) Variation of Friction with Sliding distance.](image-url)

| Table 1 – Properties of the Al-Cu-Mg alloy/4 wtBPA nano-particles composites [6,9]. |
|---------------------------------|-----------------|-----------------|-----------------|
| Density (g/cm³)                | Poisson’s ratio | Tensile modulus (GPa) | Tensile strength (MPa) |
| 2.01                           | 0.32            | 78.89            | 230.5           |
| Frequency of particles fracture | No of cycles to fatigue failures | Hardness values | Wear resistance(1/K) at 200°C |
| 0.4                            | >10⁷            | 66.8HRB          | 144.49          |


4. Conclusions

From the results and discussion above the following conclusions can be made:

1 The presence of the bean pod ash nanoparticles in the matrix alloy results in a much smaller grain size in the cast composites compared to the matrix alloy.

2 The addition of bean pod ash nanoparticles to Al-Cu-Mg alloy increases both the tensile strength and hardness values to 35 and 44.1% at 4 wt% BPA nanoparticles.

3 The impact energy decreased from 7.81 to 7.68 J at 0 and 4 wt% BPA nanoparticles.

4 The wear rate of the composites is greatly lower than the unreinforced Al–3.7%Cu-1.4%Mg alloy.

5 Toyota Carina one (1) model connecting rod was successful produced with the developed composites.

6 The fuel consumption of the developed MMCs connecting rod was lower when compared to the standard connecting rod which can resulted to 0.36% savings in fuel consumption.

7 It has been established that, replacement of connecting rod material with Al-Cu-Mg/bean pod ash nanoparticles biocomposites can results to good strength, reduced weight and induced stress in the structure with fuel saving of 0.36%.

Conflicts of interest

The author declare no conflicts of interest.

REFERENCES


