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Production of Flamboyant Pod Nanoparticles as Emerging Reinforcements for Structural Composite Materials

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Abstract. Dynamic changes in environmental conditions had necessitated continuous research towards the development of innovative structural composites with enhanced properties. To produce functional concrete-based composites, there is a need for structural modification using reinforcing fillers that could improve the strength and toughness of the composites. Agricultural exercise creates many nonedible components which constitute environmental nuisances owing to their little or no economic value. However, processing such components into useful reinforcing fillers in concrete and other matrices for composite development encourages a wealth creation from waste, environmental hygiene and ecofriendly materials for engineering applications. This study focuses only on the production of nanoparticles which can be used as fillers in composite development. Nanoparticles were produced from the flamboyant pods using a disc and optimised ball milling technique. Pods were pulverised using a disc miller and the powders obtained were ball milled at varied charge ratios and velocities to produce reinforcing nanoparticles. Particles obtained in each of the milling exercises were analysed and 2 functional interaction response surface model was developed to optimise the milling parameters and particle sizes. Result obtained indicated a decrease in size of the flamboyant pod particles as milling durations and charge ratios increase. P value of the model <0.0001 which is much less than 0.05 shows that the model is significant in explaining dependence of the particle sizes on the milling parameters. Hence, the developed model affirms that a minimum size of 3.5209 nm of the flamboyant pod particles can

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be obtained without agglomeration of particles when milling at 10 charge ratios for 17 hours 29 minutes.

Introduction

Comminution is the breaking down of solid materials into smaller particles using mechanical force. It is an important stage in many industrial processes, which includes mining, ceramic processing, and food processing [1-4]. Besides the size reduction, it helps in liberating valuable minerals from ore. The goal is to reduce particle size and increase surface area for subsequent processes like chemical reactions as found in ceramics processing, food processing, metal extraction, and composite development [5]. Comminution involves two stages which are crushing and milling. Crushing is the first stage of the comminution process. It involves breakage of large fragments into smaller pieces. Crushed powders are subjected to an additional size reduction by the mechanical milling process [6]. Millings are classified as wet, dry or cryogenic [7]. Dry milling is the breakage of particles in the air environment. It is applied to break brittle materials into pieces without suspending the feed materials or powder in any medium. Wet Milling is done in liquid medium. It is more effective than dry milling as it keeps particles together and prevents their suspension in air unlike the dry milling. It is used in breaking soft material when the dry milling is ineffective to break the powder. Cryogenic milling is carried out in inert gas (e.g nitrogen, helium) environment. The extreme coldness of the inert gas environment makes the materials/powders very brittle which aid their breakages or refinement. It is applied in breaking very soft materials which are flattening instead of breakage during the dry milling [8]. Different works are reported regarding the comminution process. Two axis rotation ball mill was designed, and its geometric model was created using CREO parametric 2.0 software. The performance of the fabricated ball mill was examined by milling 53 μm sized alumina and 37 μm sized silica at 10 charge ratios. It was concluded that the particles sizes of the alumina/silica mixture decreased as the milling duration increased [9]. Al1xxx nanoparticles were produced by milling 53 μm sized Al(1xxx) containing 0.1% carbonised coconut shell particle as the process control agent. Samples obtained at 16, 46 and 70 hours were analysed. It was reported that wide variations in particle sizes were due to different ball impacts on the individual particles during the ball milling exercises. Effects of charge ratios (mass of balls to mass of the powder) milling duration on sizes of coconut shell particles were examined using gaussian function. It was declared that the finest particle size was obtained at charge ratio value of 10 while the gaussian regression function affirmed the minimum particle size at charge ratio value of 13.49 [10]. Furthermore, effect of milling durations on particle sizes of uncarbonised coconut shell were studied [11, 12]. Sizes of the coconut shell particles obtained at 16 and 70 hours of milling were determined using images obtained from scanning electron microscope (SEM) as reported in [12]. It was shown that the particle sizes of the uncarbonised coconut shell decreased with increased milling durations. Also, sizes of the uncarbonised coconut shell nanoparticles obtained at 16, 46 and 70 hours and 10 charge ratios were determined by images obtained from SEM analysis and x-ray diffractometer aided with Scherrer's equation. Both methods affirm the decrease in the particle sizes of the uncarbonised coconut shell nanoparticles with the increase in the milling duration and there are variations among the particle sizes determined from the SEM and XRD [11]. Breakage of palm kernel, coconut and periwinkle shells were studied. Powders of palm kernel, coconut and periwinkle shells of equal sizes were milled under the same conditions. It was displayed that coconut shell particles have a smallest average size after milling for 70 hours and charge ratio value of 10 [13]. In addition, production of coconut shell-based carbon nanoparticles was reported. Carbon flakes obtained after carbonisation were milled at different durations using tumbler ball mill. It was revealed that carbon flakes attained nanoparticle size smaller than those of the uncarbonised coconut shells at 16 hours of milling [14]. Comminution is also applied in food industries in processing food granules. Maize starch was milled using a planetary ball mill operating at 500 rpm for 1, 2 and 3 hours. It was reported

according to TEM and SEM examination results that there were changes in the maize granule surfaces and cavities in the central region of the distorted granules were observed in the company with disappearance of alternate growth rings [15]. Moreover, effect of ball milling on the physicochemical and functional properties of insoluble dietary fibre obtained from three types of grain bran (prosomillet, wheat and rice), which opened a broad prospect for the by-product bran with low utilization was investigated according to the report in the article [16]. It was concluded that the ball milling operation provided an effective treatment of insoluble dietary fibres by reduction of the particle size, increase of the surface area, and more exposure of the functional groups and aiding the release of polyphenols, which improves the physicochemical and functional properties of the insoluble dietary fibres [16]. Structural, physicochemical and digestive properties of wheat starch type A and type B were examined using ball milling at 10, 20, 30 40 and 60 minutes. The milled samples were examined using SEM. It was reported that more fragmented granules existed in the starch granules as the ball milling time increased and rapidly digestible components of the milled starch samples are much more than those in the control starch samples. B type wheat granule broke easily than A type wheat granule as reported in the article [16].

Flamboyant trees are flowering plants producing pods two times, every year. The pods harbouring numerous numbers of wooden-inedible seeds. Being wooden in nature, both pods and seeds are hard materials. Their hardness makes them valuable materials for modifying structures of matrices like concrete, clay, polymer or metal for composite fabrication. Uses of flamboyant seeds and pods particles have been reported in literature [17, 18] as reinforcement in polymers to engineer new composites. Those studies indicate greater strength and ductility of the composites than their pristine matrices. The studies affirm importance of the flamboyant tree products like seeds and pods, but they provide no information on optimisation of milling parameters to produce the flamboyant seed/pod particles. This study emerged to address this gap. Attempts were made in this study to produce flamboyant pod particles by varying period of milling and charge ratios. This aims to disclose particle sizes obtained at different milling parameters. Establishing an actual charge ratio and milling durations that give minimum average size of the flamboyant pod particles through experiment and modelling is one of the focuses of this study. It is expected that the mathematical model developed in this study would guide the future researchers to predict the size of the flamboyant particles and other agricultural waste having similar hardness values when milled at a charge ratio and milling duration.

Materials and Method

The material used in this study is the flamboyant pods (Fig. 1). The dried flamboyant pods were picked after drying and falling from the trees planted for ornamental purposes within the Kwara State University, Malete. Each pod was opened into two parts to recover the harbouring seeds. The pod pieces were air blown to remove the dust and crushed to produce the flamboyant pod flakes which were subject to disc grinding to transform the pod flakes to the pod powders. Powders were sorted using a set of sieves arranging in descending order of mesh sizes with the collecting or receiving pan at the bottom of the sieve set. After pouring the powder to the top sieve and covered, they were set to vibration by sine shaker for 30 minutes. The finest powders collected in the pan were subjected to millings at different conditions. Milling speed that is the drum rotation remains constant at 100 rpm for all milling exercises. In the first milling exercise, 200g of the flamboyant pod were weighed and poured into the tumbler ball mill locally fabricated at Department of Materials Science and Engineering, Kwara State University, Malete. 1,600g of hardened steel milling balls were added which equal to 8 charge ratios (1600/200). The ball mill was tightly closed to prevent air access and milling began after switching the machine on. Milling took place for 55 hours with a stop at 25 hours to take samples for analysis. In the second milling exercise, 200g of the flamboyant powders was poured into the ball mill with an addition of 2kg of milling balls which is equal to 10 charge ratios. Milling durations are the same as in the first milling exercises.

Samples taken were observed physically and analysed using XRD, SEM, and UV-Vis spectrometer.

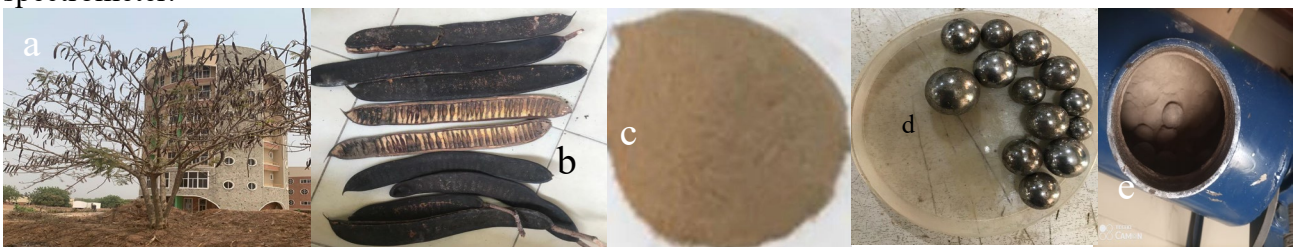


Fig. 1, Pictorial materials and milling process for producing optimised flamboyant pod particles (a) flamboyant tree (b) flamboyant pods (c) finest flamboyant powders (d) hardened stell milling balls (e) milled particles ready for discharge

X-ray Diffraction Analysis

An X-ray diffraction analysis was carried out on each milled particle to analyse the structure of crystalline materials in the particles. In the analysis X-ray beam was directed at the particles and the scattered intensities as a function of the positions (2theta) were measured. Once the beam is separated, the scatter (diffraction pattern) indicates the sample's crystalline structure. Before the analysis, particles were pressed into the sample holder at an angle of 45˚. Scanning was carried out at 25˚C in a continuous mode from angle 6˚ to 75˚(2θ) at a step size of 0.0026˚ with the aid of Philip X-ray diffractometer set at 45 kV and 40 mA using Cu anode with Goniometer radius, distant focus divergence slit and irradiated length of 240, 91 and 4 mm respectively. Peaks were assigned with phases using X'pert High Score software. Sizes of crystalline phases detected by the X-ray were determined from wavelength (λ) of the Cu radiation used for analysing the particles, shape correction factor (K), full width half maximum (β) and cosine of angle of radiation (2θ) using Scherrer's formula in Eq 1.

$$Particle\ size = \frac{\lambda K}{\beta \cos(2\theta)} \quad (1)$$

Transmission Electron Microscopic Analysis

A multipurpose field emission transmission electron microscope (TEM), Model: JEM 2100F wit attached megapixel (2048 × 2048) bottom mount CCD camera was employed to analysis the flamboyant pod particles. Particles for the TEM analysis were prepared by drying the particles on a thin carbon film coated Cu grid. Magnifying images produced by electron beam transmitted through the particles at an accelerating voltage of 100 keV were captured by the attached CCD camera to give TEM images of the analysed particles. This description of the TEM analysis is according to the procedures published in the article [19].

Mathematical Analysis

To obtain optimal parameters for the ball milling, sizes of flamboyant pod particles determined from XRD using Scherrer's equation were analysed using historical approach 2 functional interaction response surface model (HA2FIRSM). The HA2FIRSM was used because its superior features of the modelling parameters among other functions like quadratic, cubic and linear functions. Both quadratic and cubic functions are aliased implying that it is impossible for each of both functions to generate model equation for the analysed data. Both linear and 2FI functions are significant but 2FI was chosen to build the model because of its higher Fishers' value. Moreover, HA2FIRSM was developed using milling durations and charge ratios as predictor/independent variables and the particle sizes of the flamboyant pod particles as responses/dependent variable. Minimum and maximum sizes of the particles were set as lower and upper limits, respectively. The model was developed at 95% confidence with α = 5% (0.05). Model diagnostic parameters

were determined to ascertain the appropriateness of the HA2FIRSM. This analysis was built according to procedures in literature [20].

Results and Discussion

X-Ray Diffraction Diffractograms of *Flamboyant* Pod Particles

Phases of flamboyant pod particles vary as it is evident in Fig. 2a that shows different phases matched with peaks at different positions (20). The phase differences could be attributed to chemical interactions among phases of the particles during ball milling process. Similar observation was made literatures [2, 4, 11]. Differences in phases could be responsible for colour variations observed with the particles as milling durations and charge ratios increased. Superimposed curves in Fig. 2a compares the diffractograms of the milled particles obtained at 8 and 10 charge ratios by milling for 25 and 55 hours. It is observed that intensities of the milled particles are much greater than that of the control flamboyant pod power sample. This can be explained by breakages of individual powders during milling process to produce many smaller fragments. Discrepancies in the intensities of the milling particles is also clear. This testifies to the fact that the milled particles at different charge ratios and milling durations have varied sizes. Powders used as feeds for the ball milling has an average size of 37 μm. Sizes of milled particles obtained at different milling parameters, determined using Scherrer's formula in Eq 1 are presented in Fig. 2b. This shows a plot of average sizes against milling durations at different charge ratios. It indicates that the average size of the flamboyant pod particles decreases drastically at 8 and 10 charge ratios by milling for 25 hours. The smaller average size of the milling at 8 charge ratios than that at 10 charge ratios at same milling duration of 25 hours can be attributed to the fact that minimum possible size has been attached before 25 hours in the case of milling at 10 charge ratios.

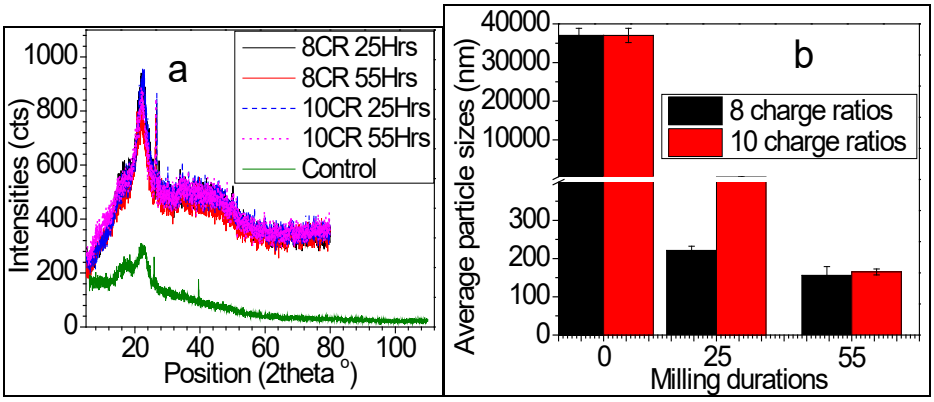


Fig. 2 , (a) superimposed diffractograms of flamboyant pod particles obtained at different milling at 8 and 10 charge ratios by milling for 25 and 55 hours (b) average sizes of the milled flamboyant particles determined using XRD aided with Scherrer's formula

Attainment of the minimum possible size is such that further milling at 10 charge ratios would result in both particle agglomeration of the finest particles due to Van der Waal's attractions and further milling of the fine particles which results in the greater average size of the particle obtained at 10 charge ratios. This explanation agrees with previous report in literature [10]. However, as milling progresses further beyond 25 hours, further particle reduction and agglomeration of ultra-fine particles accounting for slightly smaller size of the particles obtained at 10 charge ratios by milling for 55 hours than that at 10 charge ratios by milling for 25 hours. This observation is affirmed by the superimposed curves in Fig. 2a, showing much smaller intensities for the control sample. This implies that, there is no appreciable reductions in size for additional 30 hours of milling from 25 to 55 hours.

Mathematical Model

Probability (Prob>F) of the developed HA2FIRSM is <0.0001 which is smaller than α = 0.05 (Table 1). This implies that the model (see Eq 2) is significant, and it is appropriate to explain relationship between the response (particle size) and the predictor variables (milling durations and charge ratio). Both predictor variables are significant because of their probability value <0.0001. Moreover, negative beta values of both variables predict smaller particle sizes as periods of milling and charge ratios increase whereas their product has positive beta values implying increases in particle sizes as the product of the predictors increases. This explains the greater average sizes of the particles obtained at 10 charge ratios than that obtained at 8 charge ratios as the milling progressed. Therefore, the model establishes particle agglomerations which is attributed to Van der Waal's forces. It implies that the interaction of the predictor variables causes drastic particle size reduction during ball milling to produce extremely fine particles which later resulted in physical fusion of the finest particles due to their high surface area. Greater beta value of the charge ratio compared with that of milling duration is a clue supporting its higher unique contribution to refinement of the flamboyant pod particles than that of the milling durations. Furthermore, regression coefficient of determination (R^2) of the developed HA2FIRSM is 0.91228. It testifies to goodness of the model due to closeness of its value to 100%. This affirms capacities of the model to account for 91,228 data out of every 100,000. The remnants account for the residual. High Fishers' value establishes that there is a very low chance for an error to occur in using the model for predicting flamboyant particle sizes at any values each of the milling durations and charge ratios using formula in Eq 2. Adequate precision is 97.61571. This testifies to the model's ability to navigate space. Result of model's diagnosis using leverage, residuals, outliers and Cook's distance identifies a data which is out of value range among the examined 62 inputs into the software. According to Thumb's rule, one out of 62 has nothing to do with the model. Therefore, there is no serious case that demands special attention to the model. Hence, the model is good to predict the sizes of flamboyant pod particles. Moreover, optimisation of the model using minimum particle size of the flamboyant pod particle as the target goal proposes that when milling at 10 charge ratios, maximum duration of milling to obtain minimum possible size of 3.5209 nm of the flamboyant pod particle is 17 hours 29 minutes to prevent the fusion of the finest particles which can cause particle agglomeration which can result in increasing average sizes which opposes the particle refinement which is the main purpose of the ball milling operation.

Table 1, Analysis of variance

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	R^2	Adjusted R^2	Adequate precision
Model	1.23E+09	3	4.09E+08	201.0645	< 0.0001	0.91228	0.907743	97.61571
A	2.98E+08	1	2.98E+08	146.2325	< 0.0001			
B	4.72E+08	1	4.72E+08	232.1477	< 0.0001			
AB	4.25E+08	1	4.25E+08	208.8267	< 0.0001			
Residual	1.18E+08	58	2034788					

$$p = 34311.13 - 3685.36\,A - 710.901\,B + 77.0315\,AB \quad (2)$$

p = Particle size; A = Charge ratio; B = milling durations

Transmission Electron Microscopic Images

Particles of varied geometries were obtained from each of the milling exercises as it is observed in Fig. 3a-d. Variation in sizes and shapes are linked to particles responses to impacts receiving from milling balls and drum's inner surface during the milling operations. The impact effect could be an attrition leading to particle wear, compression which causes breakage or shear. All result in

particle fragmentations producing angular ultra-fine particles. It is very hard to see an isolated individual particle in each image in Fig. 3 at 20 nm resolution. This affirms physical fusion of the flamboyant pod particles during milling. Therefore, all particles exist in agglomeration. This implies that both particle refinement due to ball/drum's surface impacts and fusion due to Van der Waal's forces took place during ball milling. At the onset, when all particles are coarse, refinement supersedes agglomeration, recording high degree of particle breakages but as time goes on like above 25 hours of milling in this study, effect of particle agglomeration pronounces and opposes the purpose of the milling operation. Nanoparticle sizes obtained from XRD is supported by TEM images in Fig. 3. This also agrees with the findings from the developed HA2FIRSM in this study.

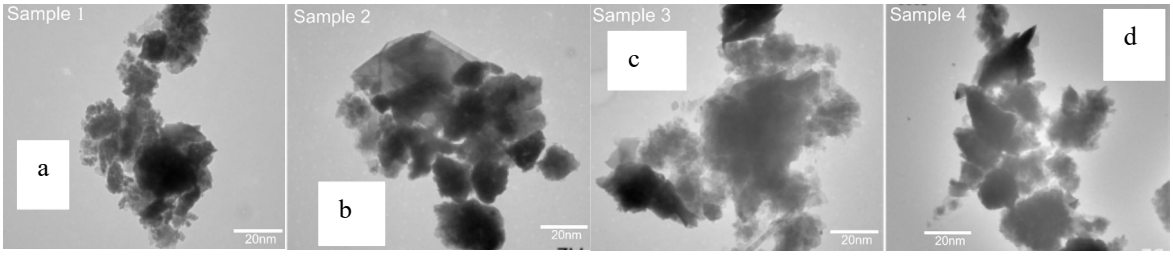


Fig. 3, Transmission electron microscopic images of flamboyant pod particles obtained at (a) 8 charge ratios, 25 hours of milling, (b) 8 charge ratios, 55 hours of milling (c) 10 charge ratios, 25 hours of milling and (d) 10 charge ratios, 55 hours of milling

Conclusions

From experimental investigation and results of this study, the following were concluded:

1. TEM indicates variation in the morphology of the particles at different milling durations and charge ratios.
2. Drastic reductions in particles sizes occurred when milling up to 25 hours.
3. Greater average sizes of the flamboyant particles obtained at 10 charge ratios could be attributed to particle agglomerations.
4. Greater beta values (coefficients of the predictor) of the charge ratio confirm its higher unique contributions to the particle refinement than milling durations.
5. Probability values of the model <0.0001 which is much less than α (0.05) affirms its significance to description of the particle size dependence on the milling durations and charge ratios
6. Moreover, optimisation of the model using minimum particle size of the flamboyant pod particle as the target goal affirms that when milling at 10 charge ratios, maximum duration of milling to obtain minimum possible size of 3.5209 nm of the flamboyant pod particle is 17 hours 29 minutes to prevent the fusion of the finest particles that may lead to particle agglomeration which can result in increasing average sizes of the particles.
7. Produced flamboyant nanoparticles are potential fillers for developing clay based or concrete based composites for building infrastructural applications.

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