



Study of optical and structural properties of natural bamboo fiber powder prepared by ball milling method

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Abstract This present work reports the preparation and characterization of bamboo powder from bamboo fibers using a ball mill technique. The bamboo fiber powder is prepared with different ball milling times of 3, 6, and 12 h. The morphology, crystallinity, functional groups, thermal analysis, absorbance, and photoluminescence of the obtained bamboo powders are analyzed. Morphological studies confirmed the decrement in the average particle size with an increase in ball milling duration. X-ray diffraction study confirms the polycrystalline nature of the synthesized powders. Particle size analysis confirms the average size of the particles 763.7 nm, 560.9 nm, and 428.9 nm for 3, 6, and 12 h ball-milled powders, respectively. The bamboo powder exhibited strong photoluminescence emission bands at 453 nm, 448 nm, and 445 nm for 3, 6, and 12 h of ball-milled powders, respectively. The blue shift observed for 12 h of ball-milled powder is due to the reduction in the size of the particles and associated enhancement of the optical band gap of the prepared bamboo particles.

1 Introduction

Bamboo finds extensive applications in day-to-day life utilities such as bamboo kitchenware, bamboo furniture, toothbrushes, and combs [<https://www.bambooindia.com>, <https://www.bambooimport.com/en/search?search=products%20from%20bamboo%209&description=1>]. The main advantage of bamboo-based products is the reduction of environmental pollution caused by the usage of plastics-based products. The bamboo products are the perfect alternative to most plastic products such as bamboo furniture, toothbrush, reusable straw, disposable platters, bamboo packaging boxes, mats, mat boards, lamps, knitting needles, doors, and dining tables [<https://www.imcorporation.in/bamboo-toothbrush.html>]. Bamboo is a common term applied to approximately 1,250 species of large woody grasses, ranging from 10 cm to 40 m in height. Bamboo is a potential natural fiber with its stiffness, rigidity, hydrophilic nature, and abundant availability. Bamboo is considered to be the second-largest resource of forestry in the world because of its rapid growth potential. Bamboo is called

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a cash crop because the time required for its cultivation is less, can be grown in deprived regions, and has a variety of uses. These fibers are cellulosic and obtained from the natural, reproducible resource of bamboo plants. Bamboo fibers are made from the pulp of the plant, which is extracted from the plant's stem and leaves. The total culm of bamboo comprises 60% parenchyma, 40% fibers, and 10% conducting tissues such as vessels and sieve tubes. Bamboo culm constitutes 60–70% of fiber content by weight. Bamboo fibers consist of cellulose, hemicellulose, and lignin in the ratio 2:1:1. Bamboo fibers are a very attractive candidate to replace artificial fibers to manufacture low-cost and eco-friendly bio-composites with tailored physical, chemical, and mechanical properties. Bamboo fibers find multifunctional applications in the clothing industry such as bamboo wearable clothes, t-shirts, kitchen towels, masks, towels, bedsheets, pillows, and kid's clothes [<https://www.imcorporation.in/bamboo-toothbrush.html>, <https://www.organicandmore.com/bamboo-fabric/><https://www.balavignaorganic.com/bamboo-fabric>]. Similarly, research on bamboo powder is also attracted a lot of attention due to the usage of bamboo powders as filling additives in many applications. The bamboo powder can be used as the raw materials of wood-plastic composites (WPC), composite plastic, outdoor decorative materials, heat insulation, moisture absorption plate, fire-resistant coating, high-grade building materials, fire-proof panels, chemical additives, plastics additive [<https://www.archiexpo.com/prod/3dwalldecor/product-66891-497904.html>]. Most of the research work was carried on bamboo powder composites such as bamboo powder/polycaprolactone, bamboo/epoxy resin, bamboo powder/PLA, and bamboo/Phenol formaldehyde (PF) resin [1–4]. Most of the studies are focused on the biological activity of bamboo powder composites but not on the characterization of bamboo powder such as morphology, crystallinity, structural, thermal, and optical properties. This research gap is explored in this manuscript by studying them. To the best of our knowledge, this is the first report on the preparation of bamboo powder using the ball milling method and their detailed characterization.

In this manuscript, the bamboo powder is prepared by ball milling method using bamboo fibers as a raw material for the first time and studied their morphology, composition, crystallinity, average particle size, functional groups, thermal properties, and photoluminescence.

2 Materials and methods

2.1 Materials

The bamboo fibers were collected from Maharashtra Bamboo Development Board (MBDB), Nagpur, India.

2.2 Preparation of bamboo powder

The bamboo fibers were initially heated at 60 °C for 4 h to remove the moisture. The bamboo powders were prepared by ball milling of the heated bamboo fibers at 300 rpm with different ball milling duration of 3, 6, and 12 h, the bamboo, and ball ratio as 1:10. The photographs of the bamboo fibers before and after ball milling are shown in Fig. 1. The obtained bamboo powder samples after the ball milling durations of 3, 6, and 12 h are named as sample A, sample B, and sample C, respectively, for easy referencing.



Fig. 1 Photographs of the bamboo fiber before and after the different ball milling durations

2.3 Bamboo powder characterization

The morphology of the powder was analyzed by a field emission scanning electron microscope (FE-SEM) (Apreo LoVac). A small amount of bamboo powder was attached to the carbon tape for the FE-SEM analysis. The crystallinity of the obtained powder was performed by X-ray diffractometer (XRD)(Bruker D8 advance). Furthermore, particle size was analyzed by a particle size analyzer (Malvern zetasizer nano S90 version 7.02). Fourier transform infrared spectroscopy (FTIR-4200) was used for the identification of functional groups present in the bamboo powders. Thermal properties were studied by thermal gravimetric analysis (TGA) (PerkinElmer STA6000). UV-1800 UV-Visible spectrophotometer is used to record the optical absorption spectra of the samples. Photoluminescence of the powders was recorded using Horiba Jobin–Yvon Fluorolog-3 (Model FL3-21).

3 Results and discussions

Figure 2a-b, d-e, and g-h shows the surface morphology of the samples A, B, and C at different magnifications, respectively. FE-SEM images of the bamboo powders show the smooth surface for all three powder samples. The decrease in grain size and increase in smoothness of the surface were observed for sample C compared to samples A and B, as expected. Furthermore, the composition of the powders was analyzed by recorded EDX spectrum. The EDX spectra of samples A, B, and C are presented in (Fig. 2c,f, and i, respectively). All the powder's EDX spectra show the prominent peaks for carbon and oxygen as major elements in their composition with a small percentage (<3%) of other elements [5]. The presence of gold (Au) peaks in the EDX spectrum was due to the gold coating of the samples. These results are strongly supported by the literature reports [6].

The XRD patterns of powder samples A, B, and C are shown in (Fig. 3a) and all the powders exhibiting polycrystalline nature. The diffraction peaks at $2\theta = 15.6^\circ$, 22° , and 34.3° were attributed to (110),(002) and (004) reflections of the crystalline structure[4, 7, 8]. Further DLS measurements were performed to measure the average particle size of the obtained powder particles. The prepared bamboo powders were dissolved in distilled water and sonicated for 30 min for uniform dispersion. (Fig. 3b–d) shows the DLS graphs of bamboo powders. The bamboo powders exhibit the average particle size of 763.7 nm, 560.9 nm, and 428.9 nm for samples A, B, and C, respectively. A decrease in particle size was observed with the increase in the duration of ball mill time (as shown in Table 1). Similar trend is reported by earlier studies [9, 10].

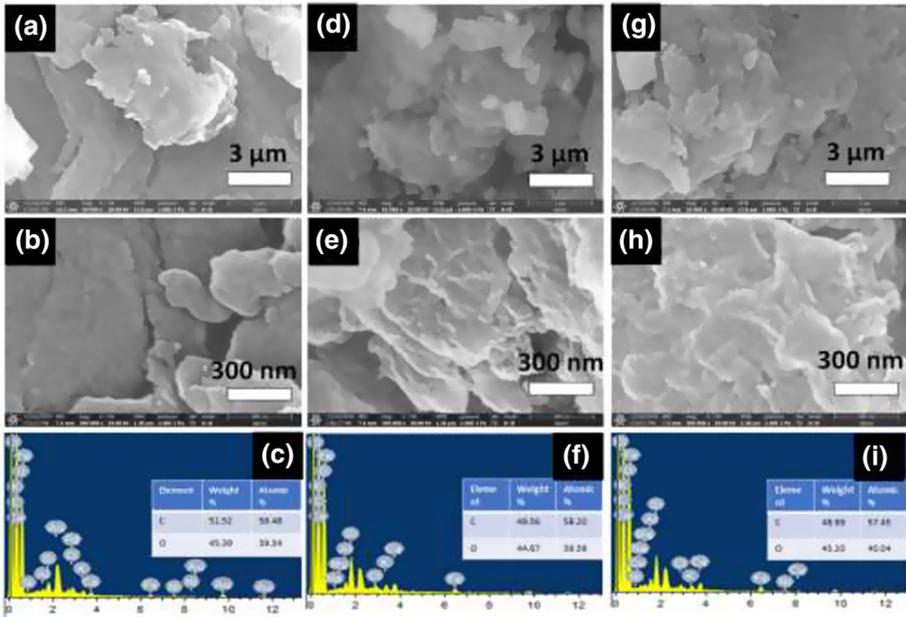


Fig. 2 FE-SEM images and EDX spectrum of the bamboo powders obtained at different ball milling durations of bamboo fibers **a–c** 3 h, **d–f** 6 h, and **g–i** 12 h

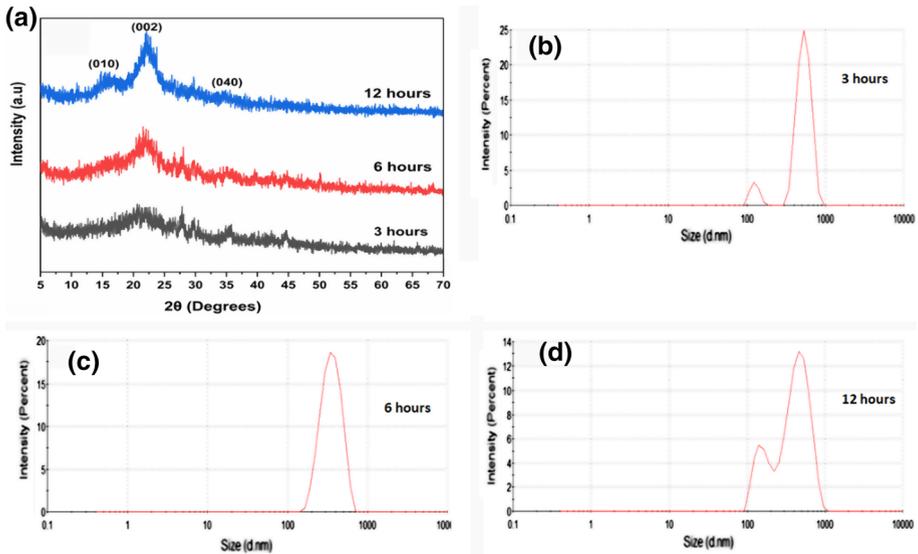
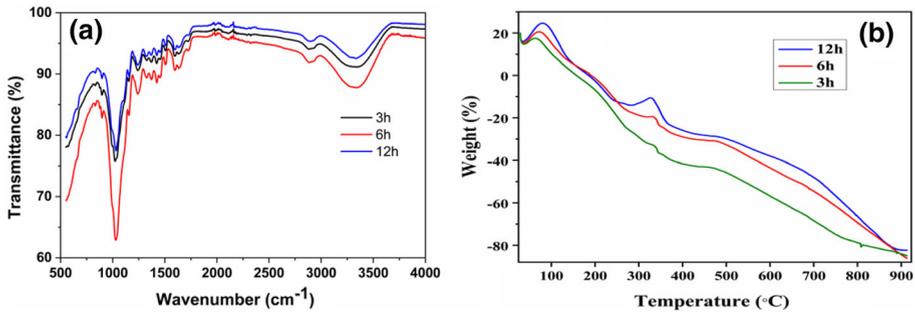


Fig. 3 a XRD pattern of the ball-milled bamboo powders, **b–d** DLS spectra of the bamboo powders

The FTIR spectrum of the prepared bamboo powders is shown in (Fig. 4a). In the FTIR spectra, the bamboo powder has two characteristic absorption peaks at the wavenumbers ~ 3410 and 2905 cm^{-1} from the stretching vibration of –OH and –CH groups. The absorption band at 1715.38 cm^{-1} can be associated with the stretching vibration of the semi-

Table 1 Optical properties of the bamboo samples

Sample	Size of the bamboo particles (nm)	Cutoff wavelength (λ) nm	Optical band gap (E_0) eV
Sample A	763.7	405	2.74
Sample B	560.9	374	2.92
Sample C	428.9	360	3.04

**Fig. 4** **a** FTIR spectra, **b** TGA spectra of prepared bamboo powders of different ball milling durations of 3, 6, and 12 h

non-conjugated cellulose ester group. The absorption peak at 1647.35 cm^{-1} belongs to the absorbed water. Moreover, the absorption peaks at 1432.56 , 1376.87 , and 1063.75 cm^{-1} represent the lignin aromatic ring bending vibration of $\text{C}=\text{C}$, the bending deformation of $-\text{CH}$, and acyclic $\text{C}-\text{O}-\text{C}$ stretching vibration of $\text{C}-\text{O}$, respectively. The absorption peak at 993.87 cm^{-1} corresponds to the characteristic peaks of fiber ether linkages, annular inner surface $\text{C}-\text{O}-\text{C}$ asymmetric stretching vibration. The absorption peak at 583.45 cm^{-1} is regarded as outer $-\text{OH}$ plane deformation vibration. All the bands are observed at the wavenumbers reported by the FTIR studies on bamboo/woven kenaf mat reinforced epoxy hybrid composites, bamboo fiber reinforced poly(lactic acid) composites and wood veneers [11–13].

TGA curves of the bamboo powders sample A, B, and C are shown in Fig. 4b. As shown in Fig. 4b, the bamboo powder has two weight loss stages, which are similar in all three curves. The first weight loss stage happens at an endothermic peak $\approx 85^\circ\text{C}$ attributed to the evaporation of absorbed water on exterior surface of the cellulose. The second weight loss stage appears at another endothermic peak $\approx 360^\circ\text{C}$ due to the absorbed water evaporation in the interior portions of the bamboo cellulose. We can clearly notice the pyrolysis of cellulose between 420 and 480°C by the decomposition of bamboo powders. More weight loss was observed for sample C compared to sample A and B. The sample C can bind more $-\text{OH}$ molecules with strong affinity, which need more quantity of heat to vanish its bound water content [14].

The optical absorption spectra of the prepared bamboo powder samples were recorded at room temperature and shown in (Fig. 5a). The cutoff wavelengths of the samples are shown in Table 1. The absorption spectrum of sample A has shown the cutoff wavelength of 405 nm . A gradual blue shift is observed in the cutoff wavelength with the increasing duration of ball milling for the samples B and C. Tauc plots are drawn by using the Tauc equation ($(\alpha h\nu)^{1/2} = C(h\nu - E_g)$) to measure the optical band gaps of the samples A, B, and C [15–17]. Tauc's plots of samples A, B, and C are shown in Fig. 5b, and the optical

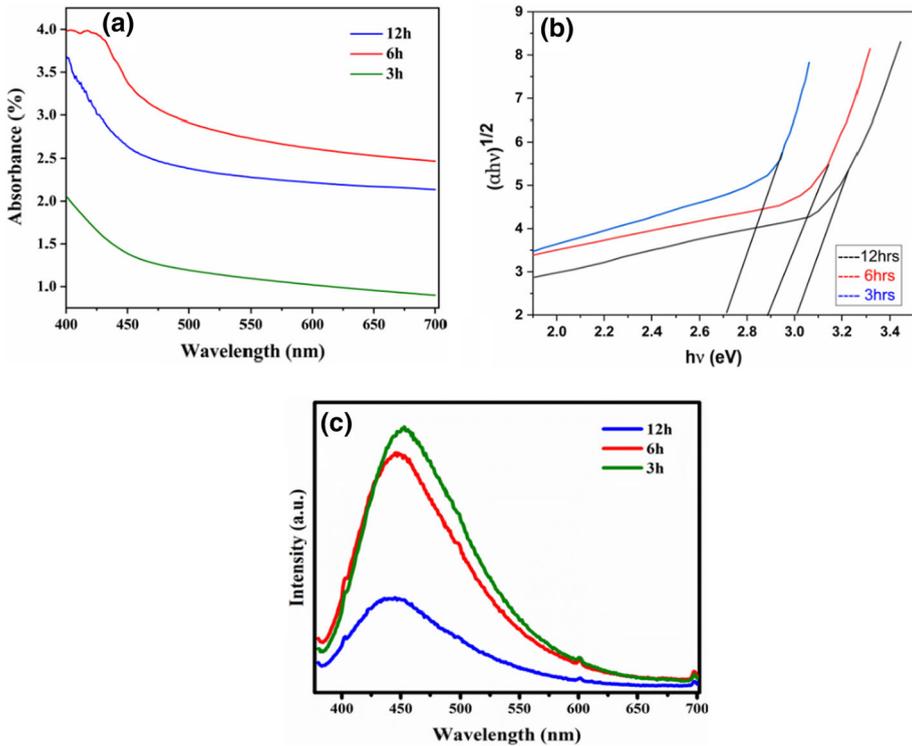


Fig. 5 a UV-Visible spectra, b Tauc plots of the prepared bamboo samples, c Photoluminescence spectra of bamboo powders

band gap (E_0) of the samples were calculated for direct allowed transitions (see Table 1). The values of the optical band gap (E^0) are observed in the order: sample C (3.04 eV) > sample B (2.92 eV) > sample A (2.74 eV). The reason for this is the size dependence of energy bands of the bamboo nanoparticles due to the quantum confinement [18]. As the grain dimension of these powders decreases then the band gap becomes broad and ultimately, the blue shift takes place in optical absorption as observed in (Fig. 5a) [19].

There are no reports available in the literature on the emission properties of the pure bamboo fiber powder so far. However, luminescence properties of bamboo-like SiC nanowires excited at 325 nm were reported by M. Zhang et al. [20]. Two wide emission bands in the range of 420–490 nm were reported. The spectra have been described in terms of the blue-shift by size confinement effect. X. Chen et al. [21] have studied the photoluminescence properties of synthesized bamboo-like 3C-SiC nanowires excited at 330 nm. They have reported wide luminescence bands in the spectra, ranging from 350 to 520 nm. The change of wavelength of emission bands was characterized in terms of particle size and band gap of the samples.

Figure 5c shows photoluminescence (PL) spectra of the prepared samples excited at $\lambda = 325$ nm at room temperature. The PL spectra show the strong emission bands at 453 nm, 448 nm, and 445 nm for samples A, B, and C, respectively. We can observe a clear blue shift in the emission maxima of the samples as a function of the ball milling time. Also, the fluorescence intensity of the samples gradually decreases from sample A to sample C.

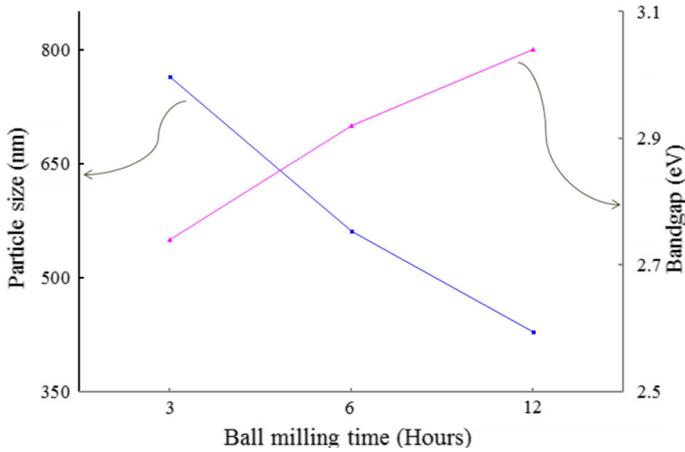


Fig. 6 Variation of particle size and band gap of the prepared bamboo powders as a function of ball milling time

Figure 5c shows the change of fluorescence intensity of the samples due to the increased compactness of the samples.

The blue shift is observed in PL spectra due to the enhancement of the optical band gap of the prepared bamboo particles. The optical band gap depends on the particle size. Variation of the band gap and particle size of the prepared bamboo powders as a function of ball milling time is shown in Fig. 6. The gradual decrease in the particle size leads to quantum confinement, which is responsible for the shifting of the emission band toward the lower wavelength side. On the other hand, we have noticed the presence of the carboxylate (COO⁻) and hydroxyl (-OH) groups in the FTIR spectra (Fig. 4a) of the samples. The gradual raise of COO⁻ and -OH groups leads to reduction of particle size [15, 22]. As the size of particles decreases, more particles come to the surface of the samples. Thus, the surface to volume ratio can be increased slowly, which leads to improvements in mechanical strength and rigidity of the samples. Therefore, the high phonon losses take place by higher vibration frequencies in sample C. Thus, the fluorescence intensity is poor for sample C. The present results will be useful in preparing bamboo powder-based products for different applications.

4 Conclusion

We have successfully prepared the bamboo fiber powder by a mechanical ball milling method with different ball milling durations. The prepared bamboo powders were characterized for their morphology, crystallinity, thermal, and optical properties. The average particle size of the powders was observed in the range of 430–763 nm. The prepared bamboo powders exhibited photoluminescence activity and strong emission peaks at 453 nm, 448 nm, and 445 nm for time durations of 3, 6, and 12 hour ball milling durations, respectively. This work offers a novel approach for bamboo powder preparation and its detailed characteristics. This study will help in designing various bamboo products for various applications.

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Author contributions NJ contributed to data collection and data analysis. LSR contributed to data analysis and interpretation. AA contributed to data analysis. TVR contributed to critical revision of the article. RRK contributed to drafting the article.

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Availability of data and material Not applicable.

Code availability Not applicable.

Declarations

Conflict of interest This manuscript entitled “Study of optical and structural properties of natural bamboo fiber powder prepared by ball milling method.” describes the original work carried out by our group. We have not published or submitted this manuscript to any other journal. All the authors of the manuscript have agreed to submit this report to The European Physical Journal Plus.

Ethical approval Not applicable.

Consent to participate Not applicable.

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