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EFFECT OF HIGH ENERGY BALL MILLING GRINDING ON PHYSICO-CHEMICAL, MORPHOLOGICAL AND OPTICAL PROPERTIES OF *CURCUMA LONGA* NANOPARTICLES POWDERS

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ABSTRACT: *Curcuma longa* nano powder was prepared by high energy ball milling equipment using green synthesis. Green synthesis is the process of synthesizing nanoparticle from biogenic resources based on the fact of its bioactive potential would be less toxic (product & bi-product), environmental safety (eco-friendly and biodegradable) cost effective and easily available to its natural origin. The powder was characterized by X-ray diffractometer (XRD), Scanning Electron Microscope (SEM), Fourier Transform Microscope (FTIR) and Photoluminescence spectrometer (PL).XRD and SEM results shows average crystalline size and grain size of turmeric powder are in nanometer range. Types of functional group and bond character in the nano powder were determined using FTIR. Optical properties of curcumin was includes changes the colour from yellow to red and emission of multicolour peak in visible region. In this approach, eco-friendly, rapid ways large amount of nano powder can be produced. Such nanometric food particles can improve the physicochemical properties of food materials. Since this materials also possess luminescence with nano size and hence may be useful in biomedical applications and pharmaceutical industry.

INTRODUCTION: Nanotechnology is the study and use of materials, devices and system that exploit arising from the structure and behaviour of matter in the nanometric size range (1 to 100nm) [1]. Nano science and nanotechnology has begun to find potential applications in the area of functional food by engineering biological molecules toward functions very different from those they have in nature, opening up a whole new area of research and development.

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These seems to be no limit to what food technologist are prepared to do our food and nanotechnology will give them a whole new set of tools to go to new extremes. But there are also a lot of positives. Lets's take a look at potentially beneficial effects nanotechnology - enabled innovations could have on our foods and subsequently on health [2]. Food is nanofood when nanoparticles, nanotechnology techniques or tools are used during cultivation, processing, or packaging of the food [3].

According to evidence the use of nanosized materials initially as crude drugs in the form of powder since thousands of years documented in various ancient scriptures based on the fact of its bioactive potential and acceptability of its to the body [4].

Turmeric as powder with its yellow colour, has been used as a dye, medicine, and flavouring since 600 BC. Turmeric used as dye their bodies as part of their wedding ritual and health benefit in India from Vedic culture. In Asian subcontinent edible turmeric have long been widely consumed as nutritional food because of their unique flavour, texture, antioxidant and functional properties.

Turmeric has a peppery and somewhat bitter taste, is deep yellowish orange in color [5, 6]. Superfine grinding by the high energy ball milling is the novel technology in food processing and can improve the physiochemical properties. Compared with other samples ground with traditional mechanical methods, superfine powder bears good physical properties like dispersibility and solubility[7]. For example, the *A. membranaceus* powder obtained with superfine grinding had high water-holding capacity, high fluidity, high water solubility index and high protein solubility [8].

In the process, the surfaces of superfine powders may change and reveal good characteristics that are not observed in bulk size materials and conventional particles, including effects of surface reactivity, small size, quantum level phenomenon, optical, magnetic, mechanical, chemical and catalytic behavior. Superfine grinding in the sub-micron range (0.1- 1μm) is increasingly significant given the development of new functional materials for various industrial applications, including biotechnology and food materials [9]. After superfine grinding, the solubility of nutritive components increases and they are easily absorbed by the human body. Researchers are investigating the benefits of turmeric for treating Alzheimer's, cancer, diabetes, arthritis, HIV, cataracts, gallstones, endometriosis, atherosclerosis, heart attacks, and more.

Several research group reported the fine powder and their possible practical applications in various chemical, Pharmaceutical and biomedical areas [10 - 11]. Ball-milling refers to the use of friction, collision, impingement, shear or other mechanical actions to modify the structure and properties of turmeric powder. Treatment of powder using ball milling is low cost and environmental friendly. The novel size reduction mechanism of E_{max} unites the advantages of different mill types, high-frequency impact (mixer mill), intensive friction (vibratory disc mill) and controlled circular jar movement (planetary ball mill) allow for unrivalled grinding performance. This unique combination is generated by the oval shape and the movement of the grinding jars. The grinding jar supports are mounted on two discs respectively which turn in the same direction.

As a result, the jars move on a circular course without changing their orientation. As a physical method of modification, ball-milling has been used to effectively decrease the relative crystallinity and increase the solubility and digestibility of powder.

Thus the present work is designed to be obtaining insight in to how the crystalline size, functional group, electronics structure, colour changes during milling of powder. Such insight may promote the uses of superfine grinding in the food science and pharmaceutical industry, which was prepared and characterized by modern scientific tools.

MATERIALS AND METHODS: Fresh rhizome of *Curcuma longa* was purchased from the market of Patna, Bihar, India and cleaned through forced running water to remove contaminant and dry at 50^0C and cut into small pieces. The dried turmeric was milled by the mixture grinder for making fine powder. Then this fine powder was used in High Energy Ball Mill (Retsch, E_{max} Germany) for ultrafine powder.

Ball milling Methodology: It is a ball milling process where a powder mixture placed in the ball mill is subjected to high-energy collision from the balls. For these experiments, we used a high energy ball-mill equipped with an insulation cover and cooling machine. The weight ratio of powder to balls (1:20) in the stainless steel jar (50 mL). Each container was filled to approximately one third of their capacity. During milling, the jars were rotated at a constant milling speed of 500 rpm for up to 5 h and 10 h respectively. The ball-milling rotational direction was changed every 30 min. The ball-milling process was carried out at 27 ^{0}C and the temperature was maintained by the air cooling system to prevent overheating. After the treatment, the samples were sealed in a bag for analysis. All the analysis was carried out in powder form.

FIG. 1: TURMERIC PLANT

RESULTS AND DISCUSSION:

Structural and Microstructural Studies: The crystalline size and structural properties of turmeric powder synthesized by ball milling were characterised by X-ray diffractometer (D8 Advance, Bruker Germany) operate at 40kV,40mA and 25 ^{0}C. The diffraction data were read in the 2θ range from 10 degree to 50 degree at 10^0/min scan rate. The sample kept on sample holder within the chamber of analytical X-ray having wavelength 1.54Å of Cu-Kα radiation. The XRD data observed for general turmeric powder, milled for 0h (general powder, prepared by kitchen based mixture-Grinder machine), milled for 5hr and 10hr are shown in **Table 1, 2** and **3** respectively, while their XRD patterns are shown in **Fig. 2, 3** and **4**. Crystalline size of different constituent were calculated using Scherrer's formula [12]

$$d = K\lambda / \beta \cos\theta \qquad \text{............ (1)}$$

Where K is a constant that has value 0.9, β (FWHM) is equal to twice the diffraction angle θ and λ is the wavelength of Cu-Kα radiation (λ = 1.54056Å). The smoothened resultant diffractograms by using the Origin 8.0 software (Originlab Corporation, Northampton, USA) and then finally calculated the relative crystallinity. The size for six most intense intensity peaks found to be between 9nm to 40 nm shown in the **Table 1, 2** and **3** respectively.

TABLE 1: XRD DATA OF GENERAL TURMERIC POWDER (0hr)

2θ of Peak (Degree)	FWHM	d-spacing	Size of particles (nm)
14.4208	0.3936	6.14226	23.53
17.5098	0.7872	5.06503	11.54
19.8975	0.6298	4.46229	14.51
22.2881	0.7822	3.98878	11.63
23.9117	0.6298	3.72149	14.60
25.8281	0.2362	3.44955	40.41
38.3717	0.9446	2.34589	10.05

TABLE 2: XRD DATA TURMERIC POWDER MILLED FOR 5hr

2θ of Peak (Degree)	FWHM	d-spacing	Size of particles (nm)
14.3064	0.7872	6.19112	11.50
17.3618	0.8659	5.10788	10.49
22.0129	0.9446	4.03801	9.679
24.0712	0.6298	3.69719	14.61
38.1942	0.9446	2.35638	10.05

TABLE 3: XRD DATA FOR TURMERIC POWDER MILLED FOR 10hr

2θ of Peak (Degree)	FWHM	d-spacing	Size of particles(nm)
14.4488	0.9446	6.13044	9.57
17.2533	0.7872	5.13975	11.54
21.9741	0.7872	4.04506	11.62
23.9583	0.9446	3.71437	9.71
038.0101	0.9446	2.36736	10.04

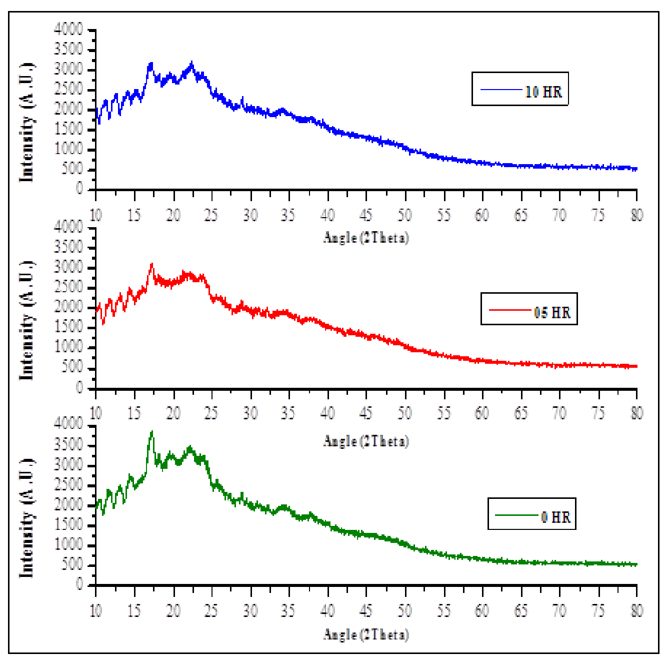
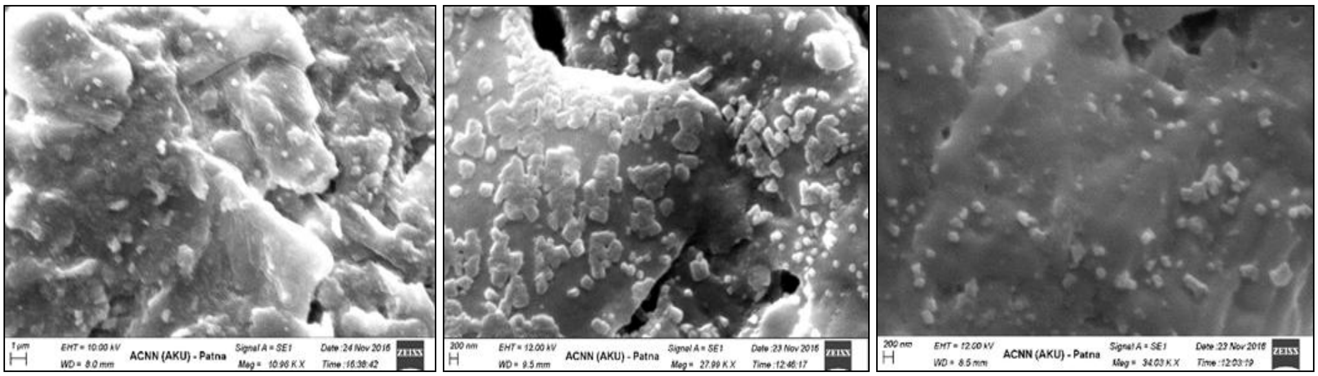


FIG. 2: XRD DIFRACTROGRAM OF TURMERIC NANO POWDER

But average particle size corresponding to prominent peak position is 11.54 nm, 10.49nm and 11.54nm respectively for three samples. It reveals that size of all constituents are in the order of nanometer size, which is also observed from SEM analysis, shown in the **Fig. 3.** Thus XRD study revealed that particle size, crystallinity and peak intensity height changes as the milling hour changes. The grain size clearly visible from SEM image. For the morphological characterization of turmeric powder

SEM (Zeiss EVO 18 Research UK) at 10kV EHT and 8.5 mm working distance. The sample was coated before used with Gold and Platinum mixture. The different intensity peak position and interplanner distance (d) also confirm the reduction of size of powder with different degree of crystallinity. Thus SEM study showed that the change in original structure of curcumin powder. This result is comparable with the other research group, who have synthesized the fine powder and found nanometric size particles [13 -14].



General Turmeric Powder **5 hr Ball milling** **10 hr Ball milling**

FIG. 3: SEM IMAGES FOR PREPARED NANO POWDER

FTIR Study: For the FTIR purpose turmeric powder were prepared by the pellet method with potassium bromide (KBr). Spectra were observed with Perkin-Elmer Model (Frontier, Thermo fisher) at room temperature. The **Fig. 4** shows the FTIR spectra of ball milled *Curcuma longa* at three different durations (a) 0 h (b) 5 h (c) 10 h. The FTIR spectra are recorded from wave number 400 cm to 4000 cm^{-1} at room temperature. In the **Fig. 4** the peak shift is not observed.

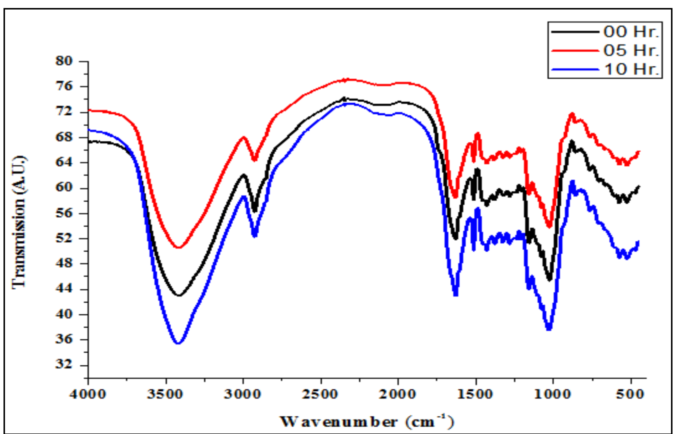


FIG. 4: FTIR SPECTRUM FOR PREPARED THREE SAMPLES

The spectrum in **Fig. 4** shows the absorbance peaks at 851, 1020, 1638, 2925 and 3412 cm^{-1}. The broad and strong absorbance peak at 3412 cm^{-1} is assigned to bonded hydroxyl (-OH) group. The absorbance peak at 1638 cm^{-1} is assigned to the carboxyl group (-C=O) stretching vibration. The assignment of all the absorbance peaks is in **Table 4**. These results show that spectrum is almost same but intensity of band was found to increase for 5hr sample and decrease for 10hr sample.

TABLE 4: FTIR DATA FOR THREE NANO POWDER

Absorbance peaks (cm^{-1})	Assignments
3412	-OH
2925	C-H
1638	C=O
1020	C-O-C
851	O-H

Optical properties: As the size and structural of a particle is changed by ball milling, the number of defects and optical properties emerges and found to be changes. Light or radiation grouped in to certain wavelength region including visible region, UV region and IR region respectively. In the **Fig. 5**, colour of the material changes from yellow to light red when the sample was milled for 5hr and finally changes to deep red after milling for 10hr. The crystalline size was also found to change as the colour of materials changes from yellow to red.

These resemble the size dependent behaviour. The important properties of luminescence spectra are judged by intensity, brightness and transition probability of an atoms or electrons. The intensity in 5hr samples are far better than 10hr samples. Milling samples for 10hr changes the particles and creates more fine particles and it may be radiation emitting atom undergoes a collision, the emission conditions change and it is equivalent to an interruption.

FIG. 5: COLOUR CHANGES DUE TO SIZE REDUCTION

The PL (Perkin Elmer LS 55, Germany) spectrum is shown in **Fig. 6** Optical properties of curcumin may be demonstrated as potential luminescent probe for the drug distribution in biological system at atomic and molecular level cell or nanoscale system. As we increase the milling hour from 0hr to 5hr then 10hr the overall intensity of the peak decreases.

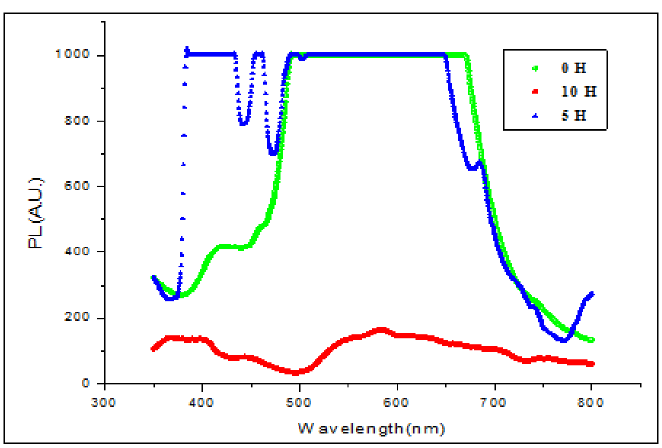


FIG. 6: PL SPECTRUM OF TURMERIC NANOPOWDER

Such appearance can be attributed due to new energy band gap between the valence band and conduction band and might be due to some intrinsic defects in the samples. Such behaviour was also observed by some research group [15 - 16]. Change in colour is associated with structural change of the materials that may lead to optical properties. Fluorescence peak intensity was found to change in all these three samples. Mai Huong *et al.*, reported that emission peak of curcumin nanoparticles shifted 540nm to 529nm after encapsulation [17]. Fang J *et al.*, reported that the 100nm particle sizes are suitable for drug targeting in tumour by enhanced permeability and retention behaviour [18]. Some research group also reported the origin of colour change in materials has been attributed to the bias induced redox switches between its cations and free radicals in organic materials [19, 20]. Various research group also prepared that nanometric size particle of curcumin, Ginger for various uses [21 - 24].

CONCLUSION: Nanometric size curcumin powder was successfully prepared by high energy ball milling equipment. XRD and SEM results shows average size and grain size are in nanometer range. FTIR spectrums are almost same but intensity of band was found to increase for 5hr sample and decrease for 10hr sample. Optical properties of curcumin may be demonstrated as luminescent source for distribution of drug at in biological system at atomic and molecular level cell or nanoscale system. From this approach, eco-friendly, faster ways large powder can be produced for different uses. Such nanometric food particles can improve the physicochemical properties of food materials. Since this materials also possess luminescence with Nanosize and hence may be useful in biomedical applications and pharmaceutical industry. Therefore superfine particles prepared by us can also be potential for drug delivery system.

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CONFLICTS OF INTEREST: There are no conflicts of interest.

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