



GLOBAL JOURNAL OF SCIENCE FRONTIER RESEARCH: D
AGRICULTURE AND VETERINARY

Volume 20 Issue 7 Version 1.0 Year 2020

Type: Double Blind Peer Reviewed International Research Journal

Publisher: Global Journals

Online ISSN: 2249-4626 & Print ISSN: 0975-5896

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GJSFR-D Classification: FOR Code: 090802



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An Experimental Study on Particle and Physiochemical Properties of Turmeric Powder Ground at Different Temperatures

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Abstract- Turmeric was ground at different grinding temperatures viz. -80°C, -40°C, 0°C and 40°C. The powder obtained at different grinding temperatures were analyzed to classify the powder. For this reason, we determined the particle size distribution, flowability, surface morphology, mineral composition and physicochemical properties of the turmeric powders at different grinding temperature. Particle size shows inverse relation with grinding temperature and, flowability shows a direct correlation with the increase in grinding temperature. We analyzed the elemental content of the samples by using SEM EDX and, the result shows that turmeric powder imparts more bioavailability of minerals with a decrease in grinding temperature. There is good retention of physicochemical properties at lower grinding temperature (-80°C) such as volatile oil (47.61%) and color (more brightness) as compared with those obtained at ambient temperature (40°C). Color parameters also significantly varied with a decrease in grinding temperature.

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1. INTRODUCTION

Spices have occupied an important place in culinary and health purposes of people since ancient times due to their characteristic flavor, aroma, color and antioxidant properties. Global spice production has increased rapidly since 1960, from 1.7-million metric tons in 1965 to 6.6-million metric tons in 2005. India is home to a variety of spices and produces around 75 types spices out of the 109 listed with ISO [1]. Turmeric (*Curcuma. long*) is one of the most studied spices because of its promising health benefits. It is known as the “golden spice” as well as the “spice of life” [2]. It belongs to the *Zingiberaceae* family, and it is mainly grown in tropical and subtropical regions [3]. India is the highest producer of turmeric as well as consumer of turmeric [4], since ancient times it is used as an Ayurvedic medicine in India [5]. In addition to this, turmeric is used in Indian cuisine as a food additive to increase shelf life and taste [6].

Few spices are used in their natural form, however, most of the spices are made into powder form

by grinding. Grinding of spices plays a key role for easy transportation, storage, mixing of components and, extraction of bioactive compounds. Curcumin is the important chemical compound present in turmeric, which is studying by many researchers. Nowadays, in food and pharma industries, there is a high demand for curcumin for its medicinal and other value addition properties. To meet the demand of curcumin in market, many industries have also started curcumin extraction units. Curcumin is present inside the cell of turmeric rhizome tissue along with oleoresin. So, before the extraction process of curcumin, turmeric rhizome needs to be ground into powder form. It is reported that finer the particle size greater the extraction efficiency for any bioactive compounds [7]. Since finer particles have more surface area, that is in contact with solvent during extraction [7]. However, a higher amount of mechanical forces is required to achieve the fine particle size, resulting in higher energy requirement and heat generation that is harmful to the flavoring, nutritional and medicinal properties of the spices [8][9]. These hindrances of the conventional grinder can be eliminated by using a cryogenic grinding technique. Cryogenic grinding is an improved version of conventional grinding. It grinds the spices with the help of cryogenics like liquid nitrogen (LN₂) [8] and maintaining an inert atmosphere inside the grinding chamber [9]. It is reported that by using cryogenic grinding, there is an improvement in the yield of volatile oil, color, good texture, uniformity of powder spices [13]. There are also a reduction in air pollution and no risk for overheat as of cryogenic grinding as compared with conventional grinding [10].

Flowability has a vital significance in handling and processing operations like storage, transportation, mixing, compression and, packing. At the industrial level, poor flowability leads to an increase in wastage and, machinery problems. Bulk density, Hausner ratio (HR), and angle of repose (α) are used in industrial-scale to measure the flow ability of powder to design the industrial equipment, storage, and transportation requirements. Moisture content and water activity of the powder plays a crucial role in shelf life and storage. Color, minerals, and volatile oil are the important properties that influence the various properties like flavor, taste, etc. during grinding. To retain various

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properties of spices, a novel grinding method like cryogenic grinding is required.

Few reports are available in the literature on the effect of grinding temperature on quality of the ground black pepper [11], fenugreek [12], king chili [13], and coriander powder [14]. However, the effect on grinding temperature on the quality of the turmeric powder was not reported yet. Therefore, the main objective of the present work is to study the changes in the particle size, particle distribution, flow ability, and physicochemical properties of turmeric ground at different grinding temperatures (-80°C to 40°C). Knowledge in the various changes in turmeric powder during different grinding temperatures helps to optimize industrial operations like storage, extraction, transportation, mixing, compression, and packing. In addition to this, the design of equipment like feed, hopper, conveyors, storage, and materials to be handle can be easily estimated.

II. MATERIALS AND METHODS

a) Sample preparation

Dried turmeric (*Curcuma longa* L.) rhizomes of Pragati variety were collected from the Indian Institute of Spices Research (IISR) Calicut, Kerala, India for, this work. Only good quality and mature rhizomes were randomly selected. The moisture content of the turmeric samples was determined by the oven drying method (Entrainment, US ISO 989) [8][15] and was 7.21% wet basis. The sample was maintained at constant moisture content by sealing in moisture resistant polythene and stored at 4 ± 1 °C. Before starting each grinding experiment, the samples kept at refrigerator temperature were brought into ambient conditions. The weighed turmeric rhizome samples (250 g) were used for grinding.

b) Grinding

The cryogenic grinding system, which consists of hammer mill with outlet sieve openings of 250 µm (ASTM-E11 60), screw conveyer type pre-cooler to get the desired temperature of the fed sample, vibratory feeder to maintain the feed rate into the pre-cooler and pressurized Dewar to pump liquid nitrogen for cooling the sample. Fig. 1 shows the schematic view of the cryogenic grinding system. The speed of the hammer mill was adjusted to 1000 rpm for all the experiments. Digital thermocouples with an indicator of ± 0.01 °C accuracy were used for measuring the temperature by putting at feeder and hopper of the grinder. The sample was fed at the rate of 40 g/min into the grinder. The grindings were done at four different regular interval temperatures viz. -80°C, -40°C, 0°C and 40°C (ambient temperature) for the testing of turmeric powder. Each experiment was replicated three times. The turmeric powders sample was stored at 4°C in moisture resistant polythene till they were analyzed for particle size

distribution (PSD), flowability, and other physicochemical properties.

c) Analysis of particle, particle size distribution (PSD) and specific surface area

Laser diffraction particle size analyzer (Fritsch GmbH Analysette 22; MicroTech Plus) was used to measure particle size, PSD, and specific surface area of the different powder. It can continuously measure particle size with a range from 0.08 to 2000 µm. For control and data evaluation, Laser diffraction particle size analyzer was connected to a computer. The computer was operated with FRITSCH MaS control software. Fraunhofer theory, a preprogram theory to Mas control was selected for calculating statistical parameters like mean diameters, specific surface area, etc.

d) Surface topography, PSD and mineral composition of the sample

To examine surface topography and mineral composition of turmeric powder Scanning Electron Microscope (SEM) (Model EVO 60; Carl ZEISS SMT, Germany) was used. Before starting the investigation, turmeric powder was coated with palladium alloy by using sputtering equipment (SC7620, England) to make sample conductive. The characteristics like surface roughness, regularity, shape, and size of the particles can be analyzed by using a software called SmartSEM. Mineral compositions of turmeric powder were determined by using the SEM-EDX technique, which is associated with a software INCA PentaFET × 3 (Oxford Instrument, UK). The samples were measured at different magnification of 250X, and 500X operated at an accelerating voltage of 20kV.

e) Flow ability measurement of powder

Hausner Ratio (HR), Carr Index (CI), and Angle of repose (α) value examined the flowability of ground turmeric sample. The following Eq. (1) [11] can define the HR of turmeric powder.

$$HR = \frac{\rho_t}{\rho_b} \quad (1)$$

where, ρ_t and ρ_b are densities of tapped and bulk, kgm^{-3} .

The bulk density was measured by pouring turmeric powder in a graduated cylinder of volume 50 ml. The upper layer of the powder was leveled with the help of an iron strip. Exact the height and weight of the powder in the graduated cylinder were measured [13, 18, 19]. The bulk density of the turmeric powder was determined by using Eq. (2) [11].

$$\rho_b = \frac{M}{V} \quad (2)$$

where, M is the weight of powder in Kg and V is Volume of powder in m^3

The tapped density was measured by manually tapping the powder in the graduated cylinder [13, 19]. The tapped density of the turmeric powder was determined by using Eq. (3) [11].

$$\rho_t = \frac{M}{V_t} \quad (3)$$

where, M is the weight of the powder in kg, V_t is the volume of tapped powder in m^3

Carr's index shows the compressibility of a powder. It is estimated by following Eq. (4) [11].

$$CI = 100 - \frac{100}{HR} \quad (4)$$

The α was measured as a maximum cone angle form by loosely pile powder on a circular base plate with a diameter of 0.05m. Laboratory setup for measurement of the angle of repose was shown in (Fig. 2). α value can be determined by the following Eq. (5) [11].

$$\alpha = \tan^{-1}\left(\frac{r}{h}\right) \quad (5)$$

where, r is the radius of the plate in m, h is the height of pile in m

f) Physicochemical analysis of the powders

Some properties like moisture content, water activity (a_w), volatile oil (V), the color of turmeric powder will be discussed in this section. Moisture content was measured by using a hot air oven method. a_w is measured by using a water activity meter (Rotornic hygrolab). Volatile oil is extracted by hydro distillation method for 7 hours for 100g of turmeric powder sample in 1000 ml at 85°C [16]. A laboratory setup for extraction volatile oil is shown in (Fig.3).

The chromaticity of the ground turmeric samples was measured using CIELab colorimeter (BYK Gardner GmbH). CIELab colorimeter has four colors for calibration, namely black calibrated standard, white calibration standard with a certificate, green checking reference, and finally by high gloss standard. After calibration, the measurements of L^* , a^* , b^* was done. The color of the turmeric samples was measured to find the values of ' L^* ' range 0 (luminance) to 100 (lightness) component, ' a^* ' ranges -120 (green) to +120 (red) axis and ' b^* ' (ranges -120 (blue) to +120 (yellow) axis. After shaking well, the ground turmeric sample measurement of color was done for three different replicas. Chroma value (C^*), hue angle (h) and yellowness index (YI) were calculated by using the following Eq. (6), (7), and (8).

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (6)$$

$$h = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (7)$$

$$YI = \frac{142.86a^*}{L^*} \quad (8)$$

g) Statistical analysis

The experimental results were analyzed with univariate analysis of variance using the least significant difference (LSD) of statistical package for the social science (SPSS) (version 20, IBM, USA). Statistical values were considered significant if $p \leq 0.05$. Each significant value was level with significant value of (a-d). Origin pro 2015 (OriginLab Corporation, USA) was used for graphical plotting.

III. RESULTS AND DISCUSSION

a) Mean particle diameters, particle size distribution (PSD) and specific surface area

From Table 1, it is clear that fineness of powder increases with the decrease in grinding temperature (from 40°C to -80 °C). The powder obtained at -80 °C gave a lower value of arithmetic mean diameter, median, and mode by 66.34%, 34.76%, and 50.07% respectively than powder obtained at 40 °C. This shows that the fineness of the particle depends on grinding temperature.

There is a significant ($p \leq 0.05$) increase in the specific surface area of the particle from 7145.16 $cm^2 cm^{-3}$ to 45960.50 $cm^2 cm^{-3}$ as grinding temperature decreased from 40°C to -80 °C as shown in Table 1. When the sample is in liquid nitrogen, the sample temperature falls below the glass transition temperature and then sample change from ductile to brittle state. This lowers the material's ability to resist high mechanical stress behavior as the grinding temperature decreases. Hence the turmeric sample becomes more brittle, easily broke down into small pieces and resulted in the higher specific surface area.

The particle size distributions with a distinct cumulative mass of different particle sizes obtained from different grinding conditions of turmeric samples are presented in (Fig. 4a-d). In (Fig. 4), the colored curves namely red and black curves denote two consecutive measurements of cumulative curve distribution of the powders over the given size range, whereas, the histograms represent the frequency of certain particle size. In (Fig. 2a-d), abscissa represents the particle size (μm), the right ordinate represents the frequency of certain particle size ($Q3(\times) [\%]$), and left ordinate represents cumulative curve distributions of powder ($Q3(\times) [\%]$).

From (Fig. 4a-d), it is clear that lower the grinding temperature distribution curve attains more skewness towards left (mean particle diameter is on the left), which affirms that finer powder is more at lower grinding temperature. Kurtosis increased (from 0.43 to 2.08) with a decrease in grinding temperature (from 40°C to -80°C). This shows that uniformity of particle size distribution increased with a decrease in grinding temperature. The maximum peak and leftmost skewness among the distribution can be seen at the

lowest grinding temperature, i.e., at -80°C . It can also be concluded that the reduction in grinding temperature showed a decrease in the particle size and more uniform PSD.

b) Surface topography, PSD and mineral composition of the sample

From (Fig. 5a-d), it can be identified that by lowering grinding temperature, there is an increase in fineness and uniformity. And (Fig. 5e-f), shows surface roughness increased with grinding temperature. The highest surface roughness, lowest specific surface area, and uniformity can be seen at 40°C . Hence, results from the SEM micrograph confirm the results of particle size and particle size distribution of the powder.

Macro elements like potassium, iron, manganese, chlorine, and copper were determined by using the SEM EDX analysis present in turmeric. Through the EDX analysis of turmeric powder, it is found that potassium content is highest followed by chlorine, copper, manganese, iron and zinc for all grinding conditions (40°C , 0°C , -40°C , -80°C). During the grinding process at different temperatures, the sample may subject to different oxidation and reduction processes, which may be the reason for the changes in the composition. All the elements present in the powder are less than 1% at all grinding conditions except for potassium.

It was found that the amount availability of elements in turmeric powder increased with a decrease in grinding temperature. This may be because of the increase in surface area imparts more bioavailability of minerals. Potassium has the highest bioavailability of elements followed by chlorine, Manganese, and Iron. Trends can be seen in Table 2., which shows that mineral composition depends on particle size. In (Fig. 6) shows the position of EDX and spectrum of turmeric powder ground at different grinding conditions.

c) Flow ability measurement of powder

From Table 4, it is clear that HR, CI, and α were increased with a decrease in grinding temperature. By comparing Table 3 and 4, HR, CI and α powder obtained from the ambient grinding (40°C) condition can be assumed as good flow characters, and remaining were having fair flow character. In (Fig. 7 & Fig. 8) shows the variation of flow ability properties (Hausner Ratio and Angle of repose) with respect to grinding temperature.

The increase in flow ability with decrease in grinding temperature could be by following reasons: a) with decrease in particle size there may be increased in the force of attraction like Van der waals force between the particles b) Moisture content, volatile oils, curcuminoids, and others mineral composition of the particles are higher at lower grinding temperature, these may an increase the cohesive forces among the particles.

d) Physicochemical properties

In Table 5, moisture content, water activity, volatile oil, and color are compiled and presented. The moisture content and water activity increased by 18.87% and 5.72%, corresponding to the grinding temperature of -80°C to 40°C respectively. An increase in the moisture content of the samples may be because of condensation of moisture present in the grinder environment. Shelf life and stability of the food sample decreases with the increase in biochemical reaction and microorganism action on the sample. Biochemical reaction and microorganism action on the sample increases, if water activity is more than 0.6 [17]. Hence, the powder obtained from different grinding conditions are stable and has a longer shelf life due to water activity is less than 0.6.

Volatile oil yield at -80°C was relatively higher as compared to that of powder at 40°C . Volatile oil yield at -80°C was 47.61 % higher than that obtained at 40°C (Table 4). Here it is clear that volatile oil yield depends on grinding temperature significantly as yield decreases with an increase in grinding temperature. Decreased in volatile oil with an increase in grinding temperature can be explained by the following facts. a) Volatile oil may be evaporated at the higher grinding temperature [9][22], b) with an increase in specific surface area, there is an increase in contact surface of powder with solvent (water) during hydro distillation extraction of oil. Therefore, the mass transfer of volatile oil from powder to water will increases [18]. Hence there is a relatively higher yield of volatile oil in cryogrinding as compare to ambient grinding.

The grinding temperature also plays an important role in producing the good color of the turmeric sample. From Table 6, it can be observed that Chroma value, Hue angle and Yellowish index of the powder produced at -80°C were 12.29%, 2.72%, 1.56% and 7.95% respectively higher than that obtained at 40°C . L^* and a^* were not varied significantly with grinding temperature whereas b^* significantly varied with grinding temperature. The color quality of the sample was good with the decrease in grinding temperature, which may be because of the reduction of mechanical forces, heat generation, and burning on the sample at lower grinding temperature. Moreover, changes in color quality may be because of change in particle size [19]. With decrease in particle size the bandwidth also decreases which results in an increase in brightness and yellowness of the particle.

IV. CONCLUSIONS

It can be concluded from the above study that grinding temperature greatly affects the quality and quantity of the powder obtained. Finer particle size, larger specific surface, uniform distribution, good color retention, volatile oil yield and mineral composition of

powder are on the side of lower grinding temperatures. It was observed that finer the particle size has significant effect on the flow ability of the powder. This paper will give the ideas about the design of grinding parts (like feed, hopper), determination of grinding temperature for better shelf life (for storage), maximum extraction (for volatile oil), excellent color (as per consumers), and higher mineral compositions.

Nomenclature:

T: Grinding Temperature (°C);

mc: moisture content (%);

a_w: water activity;

ρ_b: Bulk density (kgm⁻³);

ρ_t: Tapped density(kgm⁻³);

HR: Haunser Ratio;

CI: Carr Index;

V: Volatile oil (ml(100g)⁻¹)

α: angle of repose(Degree)

*C**: Chroma Value

YI: Yellowish index

h: Hue angle

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