Characterization of white and red teff grains using X-ray technique

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This study presents X-ray studies to investigate effect of microwave radiation and solvent treatment of white and red teff grains normally grown in Ethiopia. Studies were correlated for a better understanding of these grains.

Keywords: Microwave radiation, Solvent treatment, White and red teff grains, X-ray studies

Introduction
There is continued interest in characterization of foodgrains\(^1\). Krishnan et al\(^2\) have published on quality characteristics of biscuits prepared from finger millet seed coat based composite flour. D’Silva et al\(^3\) enhanced pasting properties of teff and maize starches through wet-heat processing with added stearic acid. Teff has been introduced in USA\(^4\) and is an annual forage crop in Michigan\(^5\). Teff is a minute, round, yellowish brown-colored grain closely resembling to millet. Teff, the Amharic word for lost\(^6\) is the name given for Teff because it is smallest grain in the world and often lost in harvesting and slashing process. It is dominant in Ethiopian diet and high in dietary fiber and iron. Teff are categorized in three main types [white, red (brown) and mixed (red, brown and white)]. White teff is favored but merely grows in some specific areas/regions of Ethiopia. It requires the most accurate growing conditions and is the most expensive type. Red/brown teff is cheapest and the least preferred type, which contains highest iron (Fe) content. It is a great source of essential amino acids, particularly lysine, which is the most deficient in grain foods. Mixed teff is hybridization of white and red teff, and has intermediate iron content. Teff contains more lysine than barley, millet, and wheat and slightly less than rice and oats\(^7\). Teff starch pellet is a compound form (diam, 2-6 µm) with multilateral shaped pellet released on milling\(^8\). Small pellet size of teff starch has considerably lower stick viscosity, higher water absorption index and lower water solubility index than maize starch\(^9\). X-ray analysis of teff starch granule gives apparently more amorphous than maize starch but similar to rice and sorghum starches in crystallinity level.

This study presents X-ray studies to investigate effect of microwave radiation and solvent treatment of white and red teff grains normally grown in Ethiopia.

Experimental Section
This study calculated cell parameters, crystallite sizes of teff from X-ray diffraction data by considering the contributions of factors to X-ray peak broadening. Structural parameters (strain & stress) were estimated by Williamson & Hall (W-H) plot. Longitudinal modulus, shear modulus, Poisson’s ratio and Young’s modulus were determined by ultrasonic pulse-echo method. Digital ultrasonic pulse echo velocity meter was used to calculate longitudinal and shear velocities. Using X-ray diffraction pattern, crystallinity of given sample was also estimated. Microwave radiation effect on these teff grains for different dosages was analyzed using X-ray diffraction data.

Determination of Teff Properties
Teff properties were determined by Central Food Technological Research Institute, Mysore. Swelling and solubility of samples were determined by reported procedure\(^10\) as modified\(^11\). Sample [500 mg, dry basis (db)] was cooked in water (20 ml) at various temperatures (50-90°C) for 30 min. Samples were weighed, made equivalent to 25 g by addition of water, and centrifuged at 3000 rpm for 15 min. Supernatant was decanted, and residue was weighed for swelling
power (SP) determination. Supernatant (10 ml) was pipetted out to a wide-mouth Petri dish and kept on a boiling water bath for evaporation. Then, dishes were dried at 105°C for 3 h, cooled, and weighed. Solubility (S) and SP were estimated as: 

\[ S = \frac{\text{wt of dry residue} \times 2.5 \times 100}{\text{wt of sample (db)}} \]

and

\[ \text{SP} = \frac{\text{wt of wet residue}}{500 - \text{wt of dried sample}} \]

Amylose was estimated according to ISO 6647 (1987). Sample (100 mg, db) was treated with ethyl alcohol (1 ml) in a conical flask (50 ml), slowly stirred, treated with 1 N sodium hydroxide (NaOH) (9 ml), and heated in boiling water for 10 min with occasional stirring. Sample was cooled at room temperature (RT), transferred to a volumetric flask (100 ml), washed, and finally made up to volume in water. A standard graph was prepared by using different proportions of amylose and starch. A standard graph was drawn with absorbance on y-axis and amylose content on x-axis. A regression equation was prepared for estimating amylose in unknown sample\textsuperscript{12}. Properties of white and red teff, respectively, were found as follows: starch, 76.0, 77.0%; amylose, 20.7, 20.2%; SP, 213.7, 204.0; and S, 4.8, 4.6.

Teff contains\textsuperscript{13}: proteins, 13.30%; total fat, 2.38%; carbohydrate (by difference), 73.13%; dietary fiber, 8.0%; calcium (Ca), 180 mg/100g; and Fe, 7.63 mg/100g. Teff flour also contained significantly more Fe than wheat flour\textsuperscript{14}. Cellulose content (6.54%) was determined by reported method\textsuperscript{15} and has been mentioned in post harvest operations\textsuperscript{16}. Protein content of teff was determined by reported method\textsuperscript{17}.

**X-ray Studies**

Raw samples of red teff and white teff grains, imported from Ethiopia, Africa, had impurities, which were cleaned manually. Some grains were powdered manually to fine powder. Some grains and powdered samples of red and white Teff were taken in a small beaker containing distilled water and kept for 2 h to prepare wet samples. This way 8 different sample (red teff grain, red teff powder, white teff grain, white teff powder, wet red teff grain, wet red teff powder, wet white teff grain and wet white teff powder) were prepared to study under X-ray Diffractometer. All samples were then placed in sample holder and kept on Goniometer. Scanning angle was varied (6 - 60°). Diffraction peaks of intensity vs \( \theta \) were recorded in computer using standard measurements. Integral breadth of diffraction peaks is related to apparent size of crystals and to their microstrains. Total strain in crystal is due to lattice mismatch between material and substrate and other crystallographic defects in crystal. If size and strain broadening is present simultaneously then crystallite size and strain are obtained from W-H plot\textsuperscript{18}, which considers both limited size of crystals and presence of crystallographic distortions lead to Lorentzian intensity distributions. Slope of plot represents average strain in crystal, whereas intercept of axis gives crystallite size as

\[ \beta \cos \theta = \frac{\lambda}{D} + 4 \varepsilon \sin \theta \]

where \( \beta \) is full width at half maxima (FWHM), D is average crystallite size and \( \mu \varepsilon \) is average strain. Plotting \( \beta \cos \theta / \lambda \) vs \( \sin \theta / \lambda \), known as W-H plot, gives a straight line with a y-intercept equal to the inverse of size and a slope equal to the value of microstrains. This method makes it possible to obtain a qualitative mean value characterizing each of the effects that cause increase in peak width. If slope of line is almost horizontal, then sample contains only a small amount of microstrains\textsuperscript{19}. 

![Fig. 1—X-ray diffraction pattern of red and white teff grains in various forms](image-url)
Further, crystallinity was also estimated and elastic modulus using ultrasonic pulse echo meter. Microwave irradiations are a non-contact, localized, rapid, uniform, energy saving and pollution free heating process. Heating materials using microwave radiations is limited to substances that can absorb this radiation and in turn dissipate this excess energy as molecular oscillations. Commercially available microwave oven (output power, 700 W), which can be varied by selection knob with marking starting from 0-100 min in steps of 10, was used. This knob denotes power%. At 10 min, power is 10% of 700, which is 70 W. Exposure to microwave radiation was carried out at 80 W for different times (10, 20 and 30 min). For calculating dosage, oven capacity was taken as 17 l, and power output was set to 385 W [385 KGY (Kilo Gray)]. Sample to oven available volume ratio is $5.85 \times 10^{-5}$. For 10 min, dosage rate is $10 \times 60 \times 385 = 231$ KGY radiation is absorbed. For 20 and 30 min of exposures, turn out to be a dosage rate of 462 and 693 KGY respectively. Samples exposed to microwave radiation were later characterized by X-ray diffraction. Parameters have standard deviation less than 5% of mean value.

Results and Discussion

Two types of teff grains have different properties. Starch content of white teff is less than that of red teff, whereas amaylose, swelling power and solubility of white teff is slightly more than red teff. Except wet white teff powder (orthorhombic) and wet red teff powder (monoclinic), crystallographic cell parameters of different samples of teff are triclinic (Table 1). Red teff is more crystalline than white teff. Powdered red teff loses its crystallinity but wet red teff powder is more crystalline.
than wet red teff grain. White teff powder is more crystalline than white teff grain but wet white teff powder is less crystalline than wet white teff grain (Table 1). Teff (both red & white) has amorphous structure (poorly crystalline). W-H plots study indicated that white and red teff contain small amount of microstrain. Ultrasonic pulse-echo study indicated that modulus is more in red teff than white teff, which is in conformity with X-ray studies that red teff has more crystalline regions to account for the strength of grain. Poisson’s ratio is found to be same for both teff samples. Microwave radiation effect showed an increase in crystallite size in red teff grains, but decrease in crystallite size for white teff with increase in dosage. There is a 10-15% change in microstructure on a broad scale in white and red teff grains due to microwave radiation, indicating there is radiation damage. In case of B-type starch crystallites, wherein a decrease in crystallite area was observed in wet starch because of less dense crystalline network packing leading to enhanced inner part of granules, which is true in present study in case of wet teff seeds.

Conclusions
Using X-ray technique, white and red teff grains have been characterized in terms of properties and behavior in presence of microwave radiation and water. Changes in microcrystalline parameters due to treatment were observed to control cooking time of teff grains.

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