

## Effect of Irradiation Doses on Quality of Cassava Starch (*Manihot esculenta Crantz*)

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### Abstract

The purpose of the study was to investigate the physicochemical, functional, and morphological properties of untreated and irradiated treated starches. Starch isolated from cassava which was exposed to gamma-irradiation at different doses of 5, 7.5, and 10 kGy. Irradiation decreased the moisture, fat, pH, and content and increased the protein, fibre, and ash content. The result revealed that the moisture content of the cassava starch ranges from 7.87-6.37, therefore the bulk and tapped density decreased ranged from 0.421 g/cc and 0.710 g/cc, respectively. Moisture content affects the flow properties of the starch. Swelling, solubility index, oil absorption capacity, and water absorption capacity increased significantly with the dose, while syneresis decreased with the dose. The Carr's Index and Hausner ratio were high as the standard values therefore according to the result the flow ability of the cassava flour was poor. Scanning electron microscopy (SEM) revealed regular, oval, polyhedral-shaped starch granules and slight shrinkage of the granules; no fissures were seen after irradiation. FTIR spectra pattern did not change by gamma irradiation.

**Keywords:** Protein, density, Shrinkage, moisture, starch

### 1. Introduction

Cassava (*Manihot esculenta Crantz*) has its origin in Latin America where it has been grown by the indigenous Indian population for at least 4000 years [1]. Tubers and roots are important sources of carbohydrates as an energy source and are used as staple foods in tropical and sub-tropical countries[2]. It has been categorized as a good source of carbohydrates due to its higher starch content (65-70%). However, freshly harvested cassava has a limited shelf life due to higher moisture content (40-70%). Thus, it will limit the utilization of the cassava itself. The drying method could increase several nutritional components in cassava, such as dietary fibre [3]. Cassava tubers have a higher starch content than other tuber crops and it was reported that the conversion efficiency of solar energy per unit land area to carbohydrates under favorable conditions exceeds that of maize and rice [4,5,6]. As a subsistence crop, it is the third most important carbohydrate food source in the tropics after rice and maize and the second most important staple food crop in sub-Saharan Africa providing more than 60% (average of 285 Cal Day<sup>-1</sup> per person) of the daily calorific needs of the populations in tropical Africa and Central America[7]. Cassava roots are a rich source of carbohydrates; most of the carbohydrates are present as starch (31% of fresh weight) with smaller amounts of free sugars (less than 1% of fresh weight). Cassava is mainly used for human consumption, industrial applications, and animal feed sectors in India. About 60% of the total cassava produced in India is used as a raw material to produce starch, sago, and dry chips; and it has scope for wider applications in food, paper, and textile industries [8,9]. It is the chief source of dietary food energy for the majority of people living in the lowland tropics, and much of the sub-humid tropics of West and Central Africa [10].

Gamma-radiation is a physical treatment in which the food is exposed to a definite dosage of rays via a free radical mechanism that hydrolyzes the chemical bonds and breaks large molecules of starch into smaller fragments of dextrans, organic acids, and sugars (Verma *et al.*, 2018) [11]. It is an ionic, fast, non-thermal, cold press, low-cost, and environmentally friendly technique requiring minimal sample preparation, no need for any catalysts as well as no pollutant agents or no penetration of toxic substances into the treated products. According to the Codex Alimentarius Commission, the irradiation technique is widely used all over the world in more than 40 countries and more than 60 different irradiated foods are available in the global market. In the current scenario, this technique is widely accepted in the international markets for its various applications in the food industries [12]. The modified starch has gained attention in the food business because of its ability to be used as a dietary fat replacer. Also, the consumption of starch within adequate limits has been linked with the prevention of different types of disease via cancer, diverticulitis, and coronary heart diseases [13,14]. It is widely employed for the enhancement of the shelf life of food products which is highly effective against all bacteria, insects, and other pests.

There have been several publications reporting on the effect of gamma radiation on various types of starches. The effect of gamma irradiation on the potato and buckwheat starch was studied by [11,15]. They revealed that there is a decrement in amylose content, pH, freeze-thaw stability, and syneresis but an increase in the swelling index and solubility power, WAC, OAC, and content. Similar results were reported by [16,17,18]. A similar result was reported by [19,20,21]. They demonstrated that decreased apparent amylose content, and pH as the dose increased on Brown rice, horse chestnut, and rice starch. The purpose of the present study was to determine the influence of gamma irradiation on the physicochemical, functional, and morphological properties of gamma-irradiated cassava starch at different doses.

## **2. Material and methods**

### **2.1. Collection of samples**

Timing cassava (*Manihot esculenta Crantz*) roots should be ready to harvest between 9 and 12 months after planting. The raw cassava was procured from the local market of New Delhi (India) for present study. The polyethylene pouches (zipper pouch) of food grade are purchased from the registered suppliers. All the chemicals used in this study were of analytical grade, procured from Merck India Ltd.

### **2.2. Starch isolation**

Fresh tubers were washed, peeled, chopped, and turn into slurry then it was diluted into distilled water at room temperature ( $22 \pm 3^\circ\text{C}$ ) for 12 h and then filtered through a 75-mm mesh sieve to separate the fiber. The starch suspension was allowed to settle for 90 min to enable the starch to settle to the bottom of the containing plastic buckets. The aqueous phase (water) was decanted whereas the sediment obtained was scrapped off from the surface and the lower white portion was recovered as starch. The wet starch was then dried in a hot air oven (Omni) at  $40^\circ\text{C}$  for 48h. The starch samples were collected and packaged in low-density polyethylene (LDPE) zipped bags/pouches and stored at ambient temperature for further analysis [22].

### **2.3. Gamma irradiation treatment**

About 500 g of dried cassava starches were packaged in low-density polyethylene bags and packed starch samples were subjected to three different doses of gamma irradiation viz, 5, 7.5, and 10 kGy. The irradiation treatments were performed at room temperature ( $25^\circ\text{C} \pm 0.5^\circ\text{C}$ ) by using cobalt-60 as a source of radiation. The irradiation treatments were performed at the Shri Ram Institute for Industrial Research, New Delhi, India. After irradiation, all samples were stored in a dry and ventilated medium to avoid any humidification before analysis.

## **2.4. Physico-chemical Analysis**

### **2.4.1.1. Proximate composition**

Moisture, ash content was analyzed as per the standard procedure given by [23].

### **2.4.1.2. Fat, Protein and Fibre**

The estimation of fat, protein, and fibre content were determined through automatic machine fat of kelplus (Pelican Equipment, Chennai, India) as described by [24].

### **2.4.1.3. Carbohydrate**

The method described by [25] was adopted to measure the carbohydrate.

### **2.4.1.4. Amylose**

The amylose content of the native and modified starch was determined using the iodine binding method described by [26]. Approximately 20 mg of starch sample was taken for analysis by using a spectrophotometer (SCHIMADZU, Japan)

### **2.5. Color characteristics**

The color values in terms of L\*, a\*, and b\* of the sample were measured using a color meter (Shenzhen 3nh Technology Co., Ltd, China) with illuminant D65 and 100 observers and the whiteness of the sample was measured by the whiteness meter (Indosaw).

### **2.6. Flow properties**

Bulk density was calculated by the method given by [27], and tap density was determined as per method given by [28]. The Porosity, Carr's Index was calculated by an equation given by [29]. Hausner ratio of the sample was calculated from the ratio of the tapped density to the bulk density [30].

### **2.7. Functional properties**

#### **2.7.1. Water absorption and oil absorption**

The water absorption capacity (WAC) of the samples (1 g) were carried out as per the method described by [31] and the oil absorption capacity (OAC) was determined by using the method as given by [32] by using centrifuge equipment.

#### **2.8. Solubility Index and Swelling Power**

Water solubility index (WSI) and swelling power (SP) were carried out at 60, 70, 80, and 90 °C with modification of sample as described by [33].

#### **2.9. Light Transmittance**

The method used for determining the light transmittance of the starch sample. 1 g was measured according to the method followed with a slightly modified the sample then the suspension was stored at refrigerated conditions (4°C) for five days and the transmittance was determined after every 24 h at 640 nm against the distilled water as blank using UV-spectrophotometer (Lasany) [34].

#### **2.10. Syneresis**

Syneresis was determined by the modified method of [12].

#### **2.11. Emulsion Activity and Stability**

The emulsion activity and stability of the sample were carried out as per the methods described by [35] and followed as the emulsion (1 g sample, 10 ml distilled water, and 10 ml soybean oil).

#### **2.12. Foam Capacity and Stability**

The method described by [36] was adopted to measure the foam capacity (FC) and foam stability (FS) of both native and modified starch.

#### **2.13. Least gelation concentration**

The least gelation concentration (LGC) was determined using the procedure recommended by [37] with modification. The flour dispersions of 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22, and 30% (w/v) prepared in 5 ml distilled water was heated at 90°C for 1 h in water bath. The contents was cooled under tap water and kept for 2 h at  $10 \pm 2^\circ\text{C}$ . The least gelation concentration was determined as that concentration when the sample from the inverted tube did not slip.

#### **2.14. Scanning electron microscopy (SEM)**

The morphological properties of the samples were observed by scanning electron microscopy. The samples were performed at the Indian Institute of Technology (IIT) in New Delhi, India. The starch granules put on a stub and gold-coated. In order to measure its morphological features double-sided sticky tape was used to apply a starch sample (1%) in ethanol on an aluminum stub. The sample was subsequently sputtered with a thin gold palladium coating and examined at an accelerating potential of 20 kV using a scanning electron microscope (ZEISS EVO 50).

#### **2.15. FTIR (Fourier Transform Infrared (FTIR) spectroscopy)**

The Fourier transform infrared (FTIR) spectra of starches were recorded on an FTIR spectrophotometer (Japan Spectroscopic Company). The starch sample was mixed with KBr and made into pellets before measurement. KBr was employed as a blank during the calibration, yielding spectra ranging from 500-4000  $\text{cm}^{-1}$ .

#### **2.16. Statistical Analysis**

Data presented for native and modified cassava starch areas means of triplicate. The Samples shall be prepared in three replication and data obtained for selected quality parameters will be analyzed for mean and standard deviations. The results were analyzed for statistical significance at  $p < 0.05$  by one way analysis of variance (ANOVA) followed by Duncan's test using IBM SPSS statistics 20 software.

### 3. Result and discussion

#### 3.1. Physicochemical properties of native and irradiated cassava starch

The proximate composition of both native and irradiated cassava starch is shown in Table 1. The moisture contents of untreated and  $\gamma$ -irradiated cassava starches were 7.87 %, 7.37 %, 6.71 % and 6.37 % for 0, 5, 7.5 and 10 kGy. Similar result showed by [38,39] as they reported that the moisture content was decreased as the doses increased in the study of lotus stem and various starch (wheat, sago and tapioca). Production of free radicals and high energy due to irradiation cleaved the glycosidic bond thus results large molecule convert into small molecules of starch. In present study, the moisture content was decreased with increasing gamma-irradiated doses due to the adsorption-desorption process. The study showed that the hygroscopicity of the cassava starch reduced due to increasing radiation doses.

The highest fat content was observed for 0 kGy was 0.47 % and the lowest was found for 10 kGy 0.19 %. A similar trend was shown in wheat and chickpea starch reported by [40,41]. The present study showed that the fat value decreased with increasing irradiation doses because of irradiation the molecular structure was changed and disrupted the double bonds of fatty acids [42].

Protein content was found in the range between 0.07 to 0.17 % from 0 to 10 kGy. The protein content were increased when the irradiation doses were increased. 10 kGy had the highest protein content whereas 0 kGy was found to be the lowest value. When the starch is treated with  $\gamma$ -irradiation it can disintegrate the non-covalent bonds (hydrogen and disulfide bond) due to the generation of free radicals which may loss of the structural and conformational integrity of the protein. The protein content was increased in wheat starch as the doses increased in the range between 0.39 to 0.40 % from 0 to 10 kGy [12].

The fibre contents were 0.20 %, 0.25 %, 0.27 %, and 0.32 % for 0, 5, 7.5 and 10 kGy respectively. It was found that the fibre content of native starch was slightly increased compared to the gamma-irradiated starch. [18] has been reported that a higher level of fibre tends to lose moisture during drying due to weak water-fiber interaction. When the fibre is present along with starch it reveals the limited amount of water available in the food system.

**Table1. Physicochemical properties of native and irradiated cassava starch.**

Parameters	Irradiation dose (kGy)			
	0	5	7.5	10
Moisture (%)	7.87±0.06 <sup>d</sup>	7.37±0.10 <sup>c</sup>	6.71±0.05 <sup>b</sup>	6.37±0.08 <sup>a</sup>
Fat (%)	0.47±0.03 <sup>d</sup>	0.35±0.01 <sup>c</sup>	0.28±0.02 <sup>b</sup>	0.19±0.01 <sup>a</sup>
Protein (%)	0.07±0.01 <sup>a</sup>	0.08±0.01 <sup>a</sup>	0.12±0.01 <sup>b</sup>	0.17±0.02 <sup>c</sup>
Fibre (%)	0.20±0.01 <sup>a</sup>	0.25±0.01 <sup>b</sup>	0.27±0.02 <sup>b</sup>	0.32±0.01 <sup>c</sup>
Amylose (%)	20.85±0.11 <sup>a</sup>	21.32±0.01 <sup>b</sup>	21.83±0.03 <sup>c</sup>	22.56±0.01 <sup>d</sup>
Ash (%)	0.057±0.001 <sup>a</sup>	0.067±0.001 <sup>b</sup>	0.068±0.002 <sup>b</sup>	0.074±0.003 <sup>c</sup>
pH	4.16±0.07 <sup>c</sup>	4.08±0.02 <sup>c</sup>	3.74±0.06 <sup>b</sup>	3.23±0.03 <sup>a</sup>
Carbohydrate (%)	91.32±0.00 <sup>a</sup>	91.86±0.01 <sup>b</sup>	92.52±0.01 <sup>c</sup>	92.87±0.00 <sup>d</sup>
Energy (kcal)	369.85±0.005 <sup>a</sup>	370.98±0.01 <sup>b</sup>	374.46±0.011 <sup>d</sup>	373.93±0.01 <sup>c</sup>

Values expressed are means ± SD (n = 3).

Means in the rows with different superscripts are significantly different (p ≤ 0.05).

The ash content for 0 to 10 kGy ranged between 0.057 to 0.074 %. Ash was slightly increased as the irradiated doses increased. Similar results were shown in  $\gamma$ -irradiated wheat starch by [12]. The composition of ash in cassava is influenced by mineral content in the soil has been reported by [43]. Ash content is a measure of inorganic impurities; it represents the total mineral contents which are used as a measurement of the quality of starch in the food industry. The pH for 0 to 10 kGy ranged in between ranges should be from 4.16 to 3.23. Similar results observed in lotus starch and wheat flour have been reported by [38,44]. Similar results were observed by [11]. Due to  $\gamma$ -irradiation, the starch molecule could be broken into carboxylic acid [31]. The present study revealed that the chemical structure of the starch molecule was changed so the pH may be slightly affected.

The amylose content for 0 kGy was found to be 20.85 % which increased to 22.56 % for 10 kGy starch. Basically, amylose content is the basis of classifying starches into waxy, semi-waxy, normal/regular and high-amylose types when amylose content is 0-2%, 3-15% 20-35%, and higher than 40% of the total starch, respectively [45,46,47]. It was found that the modified starches exhibited higher amylose content as compared to the native starch. Some studies showed that the interactions among amylose-amylopectin chains may increase as a result of irradiation. Accordingly, an elevation in the quantity of amylose chains within the granules could improve the interactions with the starch chains. As a result, the increased amylose seen with increased the doses; it may partially lead to the association between amylose-amylopectin and amylose-amylose. Increasing the amylose content by  $\gamma$ -irradiation may define the degradation of the amylopectin structures/branches and also the production of low molecular weight fractions [48,49]. Similar results for the increased amylose content were reported for corn; rice; potato; bean [50,51,52,53]. Cassava starch has been used in the production of fish crackers due to their good expansion property [39]. Amylose content is an important aspect of food processing because it affects the gelatinization and retrogradation properties.

### 3.2. Flow properties

The flow properties of native and modified cassava starch are presented in Table 2. The bulk density of the samples was observed for 0 kGy (0.554 g/cc), 5 kGy (0.557 g/cc), 7.5 kGy (0.557 g/cc) and 10 kGy (0.555 g/cc). The tapped density of a powder represents its random dense packing. The present study showed that there were no significant change in bulk and tapped density with increased doses, which indicating that  $\gamma$ -irradiation did not produce any damage to the polymer. The tapped density of the starches ranged 0.831 g/cc, 0.831 g/cc, 0.835 g/cc, and 0.831 g/cc for 0, 5, 7.5, and 10 kGy doses. This is due to some factors viz size, solid density, geometry, and surface properties of the flour material (Iwe *et al.*, 2016). Higher tapped density suits the packaging and a greater amount of material is packed within a constant unit volume [54].

The porosity of native and irradiated cassava starch ranged in between 32.97-33.24 % from 0 to 10 kGy. Porosity is a measure of number of void space present within the material. Porosity is one of the main indicators that determines the quality of bakery products and characterizes their structure, volume, and level of digestibility. The porous structure is most characteristic of bakery products and determines their quality [55].

Carr's index and Hausner's ratio are a measure of the flowability and compressibility of a powder. The Carr's percentage indicates the aptitude of a material to diminish in volume while Hausner's index shows the antiparticle friction. The values of C.I for untreated and irradiated cassava starch were ranged in between 32.97-33.24 % which was higher than the 15 % (for good flowability). It means untreated and irradiated cassava starch indicates that poor flowability. The score of Carr's index (%) of 5–10, 12–16, 18–21 and 23–28 represent excellent, good, fair and poor flowability[56]. Hausner ratio of the samples was found to be higher than 1.25 (for good flow). It seems that the Hausner ratio indicates that poor flow property of both the starches. Hausner value less than 1.25 indicates good flow and greater than 1.25 indicates poor flow [57].

**Table 2. Density and flow properties of cassava starches**

Parameters	Irradiation dose (kGy)			
	0	5	7.5	10
Bulk density (g/cc)	0.554±0.003 <sup>a</sup>	0.557±0.001 <sup>a</sup>	0.557±0.003 <sup>a</sup>	0.555±0.000 <sup>a</sup>
Tapped density (g/cc)	0.831±0.008 <sup>a</sup>	0.831±0.002 <sup>a</sup>	0.835±0.001 <sup>b</sup>	0.831±0.001 <sup>a</sup>
Porosity (%)	32.97±0.37 <sup>a</sup>	32.99±0.30 <sup>a</sup>	33.34±0.49 <sup>a</sup>	33.24±0.07 <sup>a</sup>
Carr's index (%)	32.97±0.37 <sup>a</sup>	32.99±0.30 <sup>a</sup>	33.34±0.49 <sup>a</sup>	33.24±0.07 <sup>a</sup>
Hausner ratio	1.492±0.008 <sup>a</sup>	1.492±0.006 <sup>a</sup>	1.50±0.011 <sup>a</sup>	1.498±0.001 <sup>a</sup>

Values expressed are means  $\pm$  SD (n = 3).

Means in the rows with different superscripts are significantly different ( $p \leq 0.05$ ).

### 3.3. Color Quest

The color values of native and modified cassava starch are presented in Table 3. The "L" value of the starch ranged 97.53-96.83 from 0 to 10 kG. The highest value was found for 0 kGy where as the lowest value was found in 10 kGy. The present study showed that the lightness of the cassava starch is close to whiteness.  $a^*$  value for 0 kGy, 5 kGy, 7.5 kGy, and 10 kGy was found to be -0.46, -0.31, -0.27 and -0.25 respectively.  $a^*$  value of the samples decreased as the doses were increased, which indicating the green color fell as the doses increased.  $b^*$  value ranged in between 92.96-96.43 for 0 to 10kGy. It showed that there was an enhancement of the yellow color when the doses were increased. Similar trends showed for  $L^*$  and  $b^*$  have been reported for sago starch [58], chestnut starch and bean starch [17,34]. The effect of  $\gamma$ - irradiation changed the color of starch this alteration may be due to the Maillard reaction between sugars and protein or the transformation of residual phenolics [59,60].

**Table 3. Color quest of the native and irradiated cassava starch**

Parameters	Irradiation dose (kGy)			
	0	5	7.5	10
$L^*$	94.05±0.04 <sup>d</sup>	91.58±0.03 <sup>c</sup>	85.48±0.62 <sup>b</sup>	81.40±0.52 <sup>a</sup>
$a^*$	-0.93±0.01 <sup>a</sup>	-0.64±0.00 <sup>b</sup>	-0.38±0.00 <sup>c</sup>	-0.15±0.01 <sup>d</sup>
$b^*$	9.14±0.04 <sup>a</sup>	12.12±0.02 <sup>b</sup>	12.18±0.01 <sup>bc</sup>	12.28±0.11 <sup>c</sup>
Whiteness index	82.33±0.41	79.60±0.78	78.16±0.20	74.43±0.30

Values expressed are means  $\pm$  SD (n = 3).

Means in the rows with different superscripts are significantly different ( $p \leq 0.05$ ).

### 3.4. Functional properties of native and irradiated cassava starch

#### 3.4.1. Water absorption and oil absorption capacity

The WAC and OAC of starch samples are given in Table 4. WAC for 0 kGy was found to be 1.72 % while in case of 5 kGy, 7.5 kGy and 10kGy the value was found to be (1.80 %, 1.86 % and 1.96 %), respectively. Present study revealed that the WAC increased with increased irradiation doses which may be due to the degradation of the starch into simple

sugars (like glucose, dextrin, etc.) which had a higher affinity for water than starch [20]. High WAC represents the presence of minerals, especially high phosphorus. Similar results were shown in  $\gamma$ -irradiated lotus, and broad bean starch reported by [38,34]. Similarly, [61] reported for potato starch in this, WAC increased from 84.10 to 93.24 g/g of starch on increasing the dose from 0 to 20 kGy. The high WAC is useful in the preparation of soups, and gravies and also to make food items like bread, sausages etc. WAC deals with the size, shape, proteins, lipids, pH, and salt [62], it plays a crucial role in the function of protein in various food items like in soups, bread, making dough, and in bakery products [63].

The OAC also increased with increased doses of the untreated and treated cassava starch ranging between 1.80-1.99 g/g respectively. OAC shows the binding capacity between fat and protein for the manufacturing of food products. When starch was treated with irradiation which may cause the denaturation of protein and disintegrate the physical structure of the amylopectin chain and unfolding of proteins [64,34] therefore, the starch could entrap/bind the oil. Similar results have been reported by [65,66].

### 3.4.2. Emulsion activity and stability

The emulsion activity and emulsion stability for native and irradiated starches are presented in Table 4. Emulsifying activity is defined as the maximum amount of oil that can be emulsified by a fixed amount of protein, while stability of the emulsion is defined as the rate of phase separation in water and oil during storage of the emulsion [67]. The emulsion activity for 0, 5, 7.5, and 10 kGy treated starch was found to be 26.02 %, 37.24 %, 38.79 and 50.25 % respectively with increasing doses. The emulsion stability was gradually decreased with increased doses from 0 to 10 kGy ranging between 63.07-48.14 % respectively. The present study revealed that the properties of emulsion activity and stability were changed after irradiation. The increment of the emulsion activity and the reduction of the emulsion stability might be due to the disintegration and aggregated protein molecules when the starch was treated with the dosage of irradiation [19]. Various studies have shown that a good emulsifier acts as a barrier against lipid oxidation. Emulsion properties are an important aspect in the application of food industries like the formulation of agricultural products, use in coating, etc.

**Table 4. Functional properties of native and irradiated cassava starches**

Parameters	Irradiation dose (kGy)			
	0	5	7.5	10
WAC (g/g)	1.72±0.006 <sup>a</sup>	1.80±0.003 <sup>b</sup>	1.86±0.008 <sup>c</sup>	1.97±0.002 <sup>d</sup>
OAC (g/g)	1.80±0.004 <sup>a</sup>	1.86±0.008 <sup>b</sup>	1.95±0.011 <sup>c</sup>	1.99±0.399 <sup>d</sup>
EA (%)	26.02±1.55 <sup>a</sup>	37.24±1.08 <sup>b</sup>	38.79±1.58 <sup>b</sup>	50.25±1.15 <sup>c</sup>
ES (%)	63.07±1.53 <sup>d</sup>	55.02±0.91 <sup>c</sup>	52.55±1.18 <sup>b</sup>	48.14±0.915 <sup>a</sup>
FC (%)	0.87±0.02	0.84±0.01	0.86±0.01	0.87±0.02
FS (%)	93.18±0.01	93.14±0.01	93.17±0.01	93.16±0.02
GT (°C)	62.39±0.31	62.28±0.22	62.26±0.20	62.03±0.15
LGC (%)	8%	6%	6%	4%

Values expressed are means ± SD (n = 3).

Means in the rows with different superscripts are significantly different ( $p \leq 0.05$ ).

### 3.4.3. Foam capacity and stability

The FC and FS of the native and irradiated cassava starch are presented in Table 4. The FC for 0, 5, 7.5, and 10 kGy were 0.87 %, 0.84 %, 0.86 and 0.87 % respectively. The FC refers to the surface tension and interfacial area created by whipping the protein. The FC is correlated to the protein, and the foam capacity increases when the protein is found to be high. In the current study, there is a slight decrease in foam capacity observed in 5 and 7.5 kGy treated starch. FC was found to be 93.18 %, 93.14 %, 93.17 % 93.16 from 0 to 10 kGy doses. The present study revealed that there were no significant changes after irradiation some minor changes were shown in both foam capacity and foam stability. This depends on the nature of the protein, when starch was treated with the dosage of the radiation the nature of the protein was changed which may lead to a change in the foaming properties. Similar results were shown in irradiated sorghum grains and sesame seeds by [68,69]. Foaming properties are an important aspect in the food industry and to improve the appearance or to maintain the consistency of food products such as ice cream, cake, etc. Several researchers also stated that gamma radiation caused no significant changes in foaming stability [70,66].

### 3.4.4. Gelatinization temperature and least gelation concentration

The gelatinization temperature and least gelation concentration of native and irradiated cassava starch are presented in Table 4. For untreated and irradiated starch, the gelatinization temperature should be from 62.39<sup>o</sup>C to 62.03<sup>o</sup>C. Irradiation didn't show any significant effect, it was slightly decreased as the doses increased. Gelatinization is correlated to the properties of the amylose and amylopectin chains. It deals with the loss of birefringence and crystallinity due to the breakage of the double bond and enhancing the leaching property of the amylopectin due to irradiation by [39,71,72]. It is a process where the starch granules form a suspension in cold water at a lower temperature. When heated before the storage at low temperature the cellulose wall of the starch ruptures and results in swollen bumps into each other and

absorbed water. For 10 kGy treated cassava starch had the least LGC (4 %) while 0 kGy had 8 % LGC. It was observed that the LGC was decreased as increased in doses from 0 to 10 kGy. Lower LGC had a good ability to form the gel that is used in various food items. The gelation properties of cassava starch could enhance their utilization in the food industry which is required for the formation of gel [73]. Improvement of gelation capacity will be advantageous in hastening the food product preparation [66].

### 3.4.5. Swelling power and solubility index

Swelling and solubility index of the untreated and irradiated starches is presented in Table 5. SP and SI can be used to identify the extent of bonding among amylose and amylopectin chains in crystalline and amorphous region of starch. The variation in the swelling power and solubility index with different doses of the starches as shown in Figure 1. Amylopectin plays a vital role in the swelling power; it traps the starch and retains the water within the structure, before and during gelatinization [74]. The swelling power for 0, 5, 7.5, and 10 kGy treated cassava starch ranged from 2.21-5.15 g, 2.74-5.36 g, 2.84-5.56 g, and 2.94-5.86 respectively at 50 to 90° C. The swelling power was increased with increasing temperature as well as radiation doses were increased. This result showed due to the de-polymerization of the amylopectin chain in the starch granules during irradiation and showed degradation of the starch granules chain at higher temperatures with gelatinized starch and denatured the protein matrix which may prevent the diffusion of water into the matrix of starch (Kumar *et al.*, 2017) [19]. It plays an important role in fully fulfilling the requirements of the food industry. It ascribes the classification of the starch from different botanical origins which indicate different swelling powers at different temperatures [75]. High swelling power showed the digestibility of starch is high and the usage of starch in a range of dietary applications [76]. Similar results have been reported by [20,11,77].

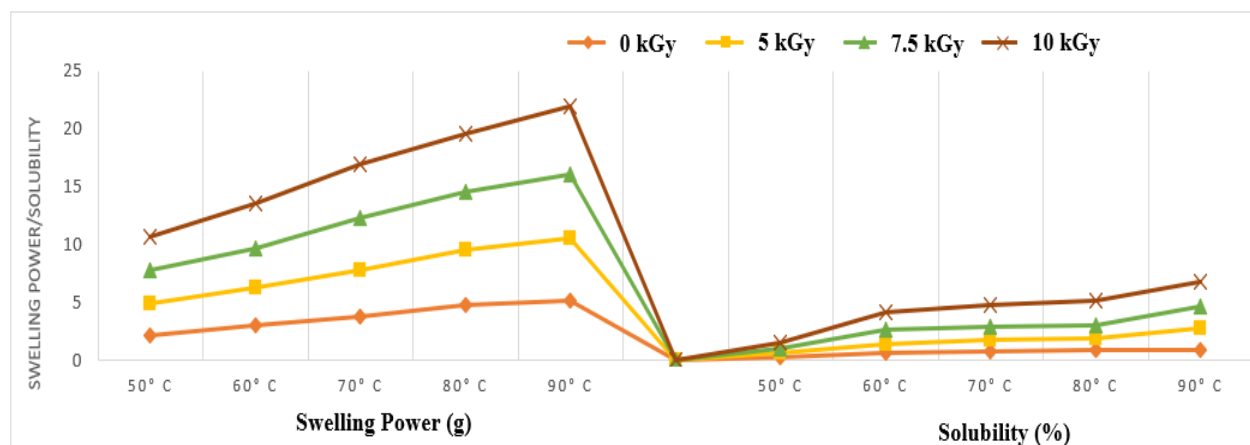
The solubility index deals with the degradation of the starch which is correlated to the amylose and amylopectin. The solubility of the samples was increased with the temperature along with doses (0 kGy to 10 kGy) ranging from 0.26-0.57 to 0.96 to 2.15 at 50° C to 90° C. Similar results for increased solubility of sweet potato, cowpea, and potato starch were demonstrated by [78,79]. Similar trends followed by [49,34]. Increased in the solubility of the starch with the amylose due to the disruption of the starch granules because there is the establishment of H-bond with water when it is released in the solution therefore, amylose content was leached out.

**Table 5. Swelling and Solubility index of native and irradiated cassava starches**

Parameters		Irradiation dose (kGy)			
		0	5	7.5	10
Swelling Power (g)	50° C	2.21±0.041 <sup>a</sup>	2.74±0.041 <sup>b</sup>	2.84±0.043 <sup>c</sup>	2.94±0.027 <sup>d</sup>
	60° C	3.04±0.023 <sup>a</sup>	3.23±0.037 <sup>b</sup>	3.43±0.027 <sup>c</sup>	3.84±0.038 <sup>d</sup>
	70° C	3.84±0.038 <sup>a</sup>	4.03±0.019 <sup>b</sup>	4.48±0.075 <sup>c</sup>	4.56±0.033 <sup>c</sup>
	80° C	4.74±0.014 <sup>a</sup>	4.86±0.027 <sup>b</sup>	4.96±0.024 <sup>c</sup>	5.06±0.027 <sup>d</sup>
	90° C	5.15±0.039 <sup>a</sup>	5.36±0.027 <sup>b</sup>	5.56±0.026 <sup>c</sup>	5.86±0.024 <sup>d</sup>
Solubility Index (%)	50° C	0.26±0.005 <sup>a</sup>	0.36±0.005 <sup>b</sup>	0.41±0.005 <sup>c</sup>	0.57±0.005 <sup>d</sup>
	60° C	0.65±0.005 <sup>a</sup>	0.78±0.005 <sup>b</sup>	1.20±0.011 <sup>c</sup>	1.55±0.015 <sup>d</sup>
	70° C	0.77±0.010 <sup>a</sup>	1.05±0.005 <sup>b</sup>	1.15±0.040 <sup>c</sup>	1.77±0.015 <sup>d</sup>
	80° C	0.86±0.005 <sup>a</sup>	1.08±0.052 <sup>b</sup>	1.14±0.046 <sup>b</sup>	2.05±0.025 <sup>c</sup>
	90° C	0.96±0.005 <sup>a</sup>	1.86±0.005 <sup>b</sup>	1.88±0.020 <sup>b</sup>	2.15±0.020 <sup>c</sup>

Values expressed are means ± SD (n = 3).

Means in the rows with different superscripts are significantly different ( $p \leq 0.05$ ).



**Fig 1. The variation in the swelling power and solubility index with different doses of the starches**

3.4.6. Syneresis

Syneresis is the separation of liquid from a gel its values depend on the storage time period. Syneresis of the untreated and irradiated starch is presented in Table 6 and was measured up to 120 h of storage. The variation in the syneresis with different doses of the starches as shown in Figure 2. Syneresis was increased during storage period for 0, 5, 7.5, 10 kGy treated starch were ranged in between 1.94-63.80 %, 1.74-61.64 %, 1.75-60.13 % and 1.34-59.04 % respectively. The highest value of syneresis was found for 10 kGy and lowest value found in untreated cassava starch at 120 h during storage at lower temperature. The syneresis value was increased with storage but decreased with the dosage. High syneresis value was found for 0 kGy ranged in between 1.94 % to 63.80 % while lowest value observed for 10 kGy treated starch ranged in between 1.34 % to 59.04 % during storage. Syneresis is the physical phenomenon in which the water is expelled and released from the starch gel [80]. Syneresis occurs due to the re-crystallization of the amylose in the granules of starch at low temperature in a loss of water from the gel structure [81]. Similar results for syneresis were reported for irradiated kithul, rice, horse chestnut starch [82,83,20]. The increase in syneresis during storage could be attributed to the interaction of leached amylose chains which result in the release of water [84,85] while decreased as the dosage increased due to the weak interaction of H-bond between amylose and amylopectin chain and to form simple sugar which have higher tendency for water.

Table 6. Syneresis (%) of the native and irradiated cassava starches

Time (h)	Irradiation dose (kGy)			
	0	5	7.5	10
0	1.94±0.02 <sup>a</sup>	1.75±0.03 <sup>b</sup>	1.74±0.03 <sup>c</sup>	1.34±0.04 <sup>d</sup>
24	55.96±0.16 <sup>a</sup>	54.55±0.42 <sup>b</sup>	53.07±0.97 <sup>c</sup>	51.77±0.51 <sup>d</sup>
48	59.50±0.18 <sup>a</sup>	56.96±0.12 <sup>b</sup>	56.29±1.08 <sup>b</sup>	55.33±1.40 <sup>b</sup>
72	59.98±0.18 <sup>a</sup>	57.92±0.09 <sup>b</sup>	57.57±0.24 <sup>b</sup>	56.80±0.25 <sup>c</sup>
96	60.80±1.84 <sup>a</sup>	60.63±0.02 <sup>a</sup>	59.50±0.17 <sup>b</sup>	58.48±0.04 <sup>b</sup>
120	63.80±0.15 <sup>a</sup>	61.64±0.56 <sup>b</sup>	60.13±0.02 <sup>c</sup>	59.04±0.06 <sup>d</sup>

Values expressed are means ± SD (n = 3).

Means in the rows with different superscripts are significantly different (p ≤ 0.05).

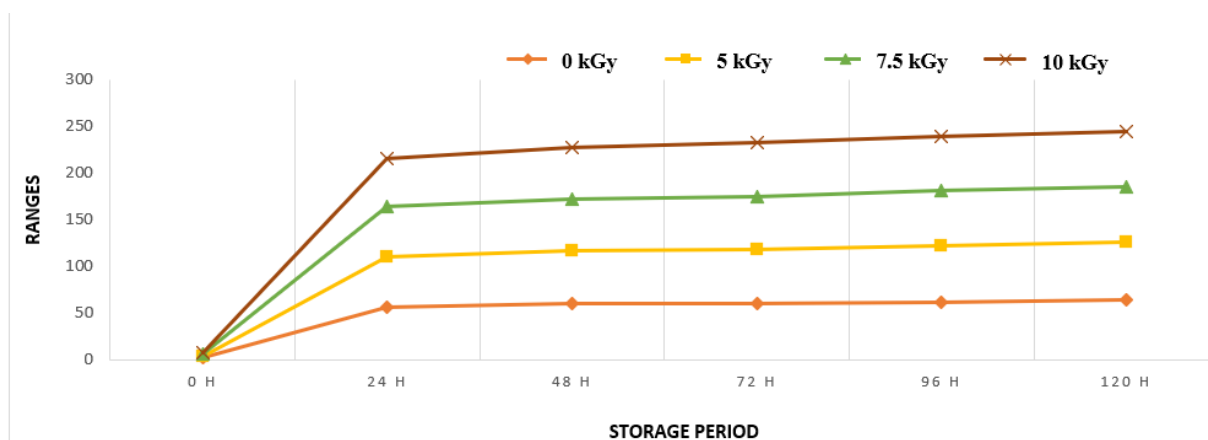


Fig 2. The variation in the syneresis with different doses of the starches

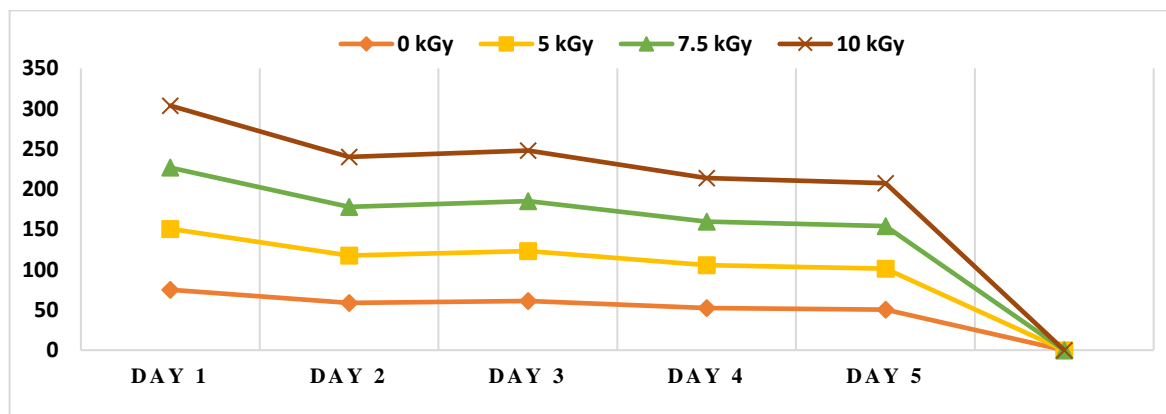


Fig 3. The variation in the transmittance with different doses of the starches



### 3.4.7. Paste clarity

The effect of refrigerated storage on the transmittance of untreated and irradiated starch gels is shown in Table 7. Transmittance is the fraction of incident light at a specified wavelength that passes through a sample. The light transmittance was decreased within the increase in storage period at refrigeration temperatures ranging between 75.06-50.17, 75.66-51.22, 76.04-52.76 and 76.98-53.09 % from 0 to 10 kGy treated cassava starch. Light transmittance was increased as the dosage increased, ranging between 75.66-76.98 %, 58.52-62.16 %, 61.24-62.89 %, 52.42-54.12 % and 50.17-53.09 kGy from 0 to 10 kGy. Similar result was observed in lotus starch, wheat starch, Indian beans, kithul irradiated starches [86,12,82]. The variation in the transmittance with different doses of the starches as shown in Figure 3. The present study suggested that cassava starch could be used in the processing of jam, jellies confectionaries food items. Transparent gels can be used as carriers of active ingredients composed of oils, surfactants, vitamins, sunscreen agents and antioxidants in the formulation of multifunctional cosmetic gels [87]. Transmittance was increased due to the disintegration of amylopectin, forming carboxyl group due to the production of free radicals, aggregation of amylose, intra-molecular bonding and swollen granules when starch treated with irradiation starch these are the factors which are responsible for improving the starch gel clarity.

**Table 7. Transmittance (%) of the native and irradiated cassava starches**

Day (s)	Irradiation dose (kGy)			
	0	5	7.5	10
1	75.06±0.01 <sup>a</sup>	75.66±0.01 <sup>b</sup>	76.04±0.02 <sup>c</sup>	76.98±0.02 <sup>d</sup>
2	58.52±0.01 <sup>a</sup>	59.16±0.01 <sup>b</sup>	60.22±0.02 <sup>c</sup>	62.16±0.02 <sup>d</sup>
3	61.24±0.02 <sup>a</sup>	61.72±0.02 <sup>b</sup>	62.09±0.02 <sup>c</sup>	62.89±0.02 <sup>d</sup>
4	52.42±0.02 <sup>a</sup>	53.17±0.00 <sup>b</sup>	53.96±0.02 <sup>c</sup>	54.12±0.01 <sup>d</sup>

Values expressed are means ± SD (n = 3).

Means in the rows with different superscripts are significantly different ( $p \leq 0.05$ ).

### 3.4.8. Scanning Electron Microscopy of cassava starches

Scanning electron microscopy (SEM) images of native and irradiated cassava starch are shown in Figure 4. In present study showed the diameter/dimension, and cross-sectional area of the granules which were calculated by using programming software image J, for untreated and treated starch granules showed same dimension of 10 $\mu$ . Some regular, oblate, round shape distribution showed in 0 kGy, this implies that during isolation process didn't cause damage, disorganize the arrangement of starch granules while the longer and elongated edges, oval, irregular and polyhedral shapes were observed in 5 and 10 kGy starches. While some granules showed cavities (see red circle) and the protein matrix was scarcely noticeable and less rough, which implies a partial solubilization which were seen in modified starch. Morphological modifications were related with starch damage during the irradiation process, as has been previously reported. Modified starch granules showed gradual loss of the polyhedral form of the starch granules was clearly visible, which implies a partial starch gelatinization. Figure 4 (a,b) shows starch granules with a smooth surface which was native, while Figure 4 (c,d) shows dispersed starch granules embedded into the protein matrix (pm) and protein bodies (pb) on the surface with fissure (see yellow circle). Clumping can be seen from the starch granules as shown in figure 4 (b, c & d) which was modified starch. This observation indicated the certain granules had been ruptured, leaching out the amylose lead to the clumping of starch granules. Greater granule swelling may exist which might leads to the granules ruptured and clumps due to effect of gamma-irradiation. Figure 4.1 & 4.2 showed the major cross-sectional area and diameter observed for 0, 5, 7.5 and 10 kGy were accounts 0.141 mm<sup>2</sup> (141.49  $\mu$ m<sup>2</sup>), 0.079 mm<sup>2</sup> (79.50), 0.131 mm<sup>2</sup> (131.30) and 0.065 mm<sup>2</sup> (65.71) and the average diameter accounts 415.0  $\mu$ m, 29.88  $\mu$ m, 41.65  $\mu$ m and 23.68  $\mu$ m respectively. As the doses increased it was observed that the shape of the granules was slightly changed it may be due to the clumping of the starch which indicate the aggregation of the starch due to the weak interaction between starch-starch molecule [88] therefore, the amylose leached out or disintegrate the physical structure of the amylopectin chain and unfold the protein matrix [64,34].

**Table 8. The native and irradiated cassava starch wave number (cm<sup>-1</sup>) is based on the FTIR spectroscopy profile observed at 4000-400 cm<sup>-1</sup>.**

S.No.	Wave number (cm <sup>-1</sup> )	Bond/ Structure	Functional group	Bond strength	Peak vibration
1.	2500-3300	O-H	Carboxylic acid	Strong, broad	Stretching
2.	2700-3200	O-H	alcohol	Weak, broad	Stretching
3.	3000-3100	C=H	alkene	Meadium	Stretching
4.	2140-2175	S-C $\equiv$ N	thiocyanate	Strong	Stretching
5.	2150	C=C=O	ketene	-	Stretching
7.	1000-1400	C-F	fluoro compound	Strong	Stretching
8.	1650-2000	C-H	Aromatic	Bending	Stretching

10.	1600-1650	C=C	conjugated alkene	Medium	Stretching
11.	1566-1650	C=C	cyclic alkene	Medium	Stretching
12.	1380-1390	C-H	aldehyde	Medium	Bending
13.	1266-1342	C-N	aromatic amine	Strong	Stretching
14.	860-900	C-H	1,2,4-trisubstituted	Strong	Bending
15.	550-850	C-Cl	halo compound	Strong	Stretching

### 3.4.9. Fourier Transfer Infrared Spectroscopy

The samples were analyzed by FTIR to confirm the breakdown of glycosidic bonds and decrease the short-range crystalline order (double helices). The FTIR spectra of the native and treated starch samples are shown in Table 8 and the spectra as shown in Figure 5. The FTIR absorption bands at  $2500-3300\text{ cm}^{-1}$ ,  $2700-3200\text{ cm}^{-1}$ ,  $1650-2000\text{ cm}^{-1}$ ,  $1380-1390\text{ cm}^{-1}$ ,  $860-900\text{ cm}^{-1}$ ,  $1600-1650$ , indicate the presence of O-H, C-H, C=C group and alcohol, aromatic, aldehyde group with weak, bending, medium and strong bond strength. The range between  $2700-3200\text{ cm}^{-1}$  that can attribute to O-H stretching in hydrogen bonds and strong inter and intra-molecular interaction between the polysaccharide chains. Gamma-irradiation can affect the peaks of the sample which showed absorption bands at:  $1014\text{ cm}^{-1}$  for 0 kGy,  $1024\text{ cm}^{-1}$  for 5 kGy,  $1829\text{ cm}^{-1}$  for 7.5 kGy,  $1843\text{ cm}^{-1}$  for 10 kGy showed the presence of C-F compound with strong bond strength and stretching peak vibration which corresponded to a aldehyde and skeletal stretching due to the aromatic ring at  $1268\text{ cm}^{-1}$ ,  $1383\text{ cm}^{-1}$ ,  $1275\text{ cm}^{-1}$ ,  $1288\text{ cm}^{-1}$ . Some showed compressed bond length with weak bands absorbed at  $593\text{ cm}^{-1}$ ,  $545\text{ cm}^{-1}$ ,  $555\text{ cm}^{-1}$  and  $553\text{ cm}^{-1}$  were ascribed to C-Cl stretching vibration. The C-H bands, which appear around  $3000\text{ cm}^{-1}$  showed organic molecules have such bonds, most organic molecules will display those bands in their spectrum which corresponding to the C=C bond stretching vibration at about  $1600-1700\text{ cm}^{-1}$  ascribed the presence of alkene group. The peak below  $900\text{ cm}^{-1}$  (finger printing region) with the major band at  $500\text{ cm}^{-1}$ ,  $550\text{ cm}^{-1}$ ,  $600\text{ cm}^{-1}$ ,  $650\text{ cm}^{-1}$  and  $750\text{ cm}^{-1}$  represent pyranose ring in the glucose unit of starches [12,90].

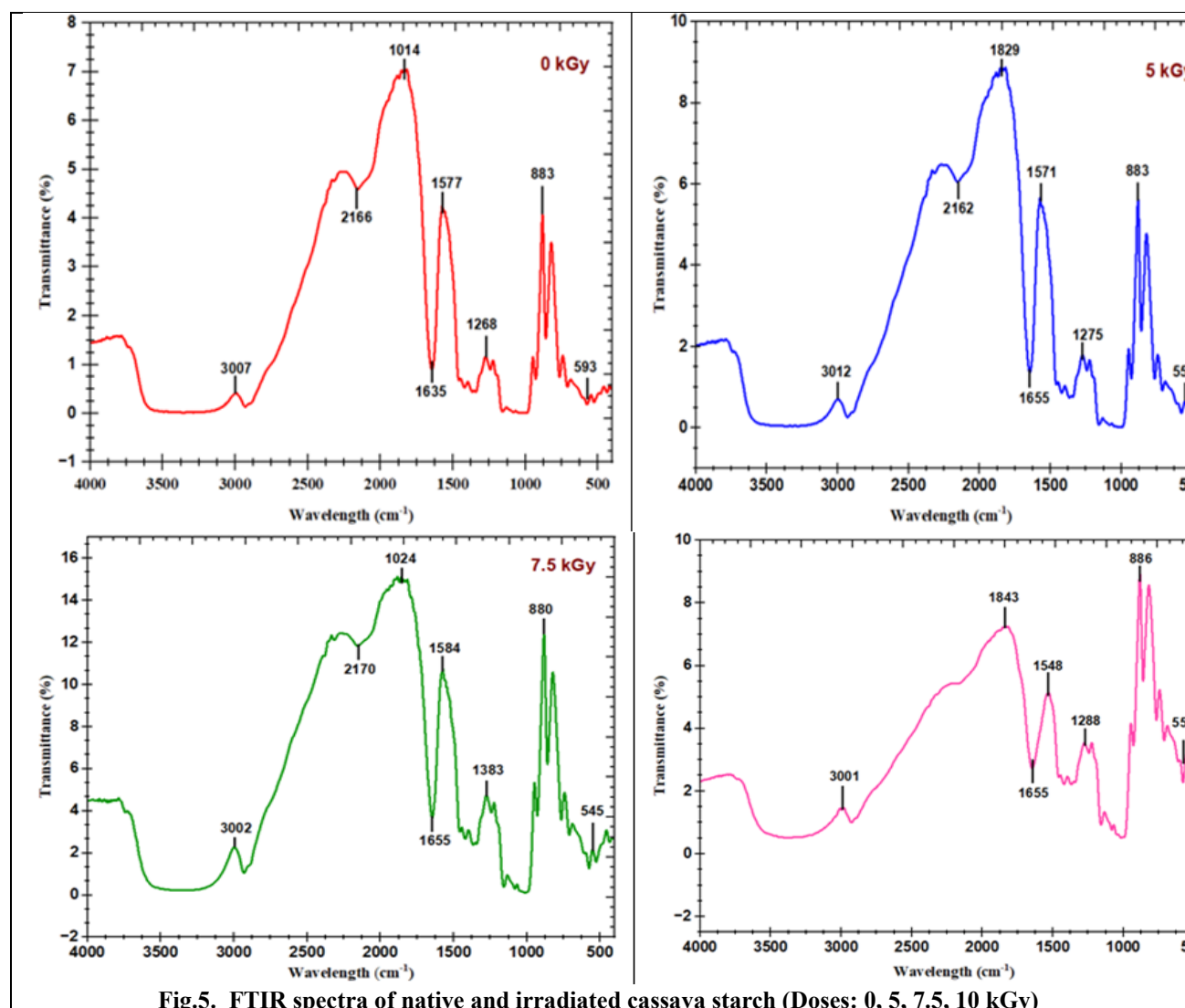
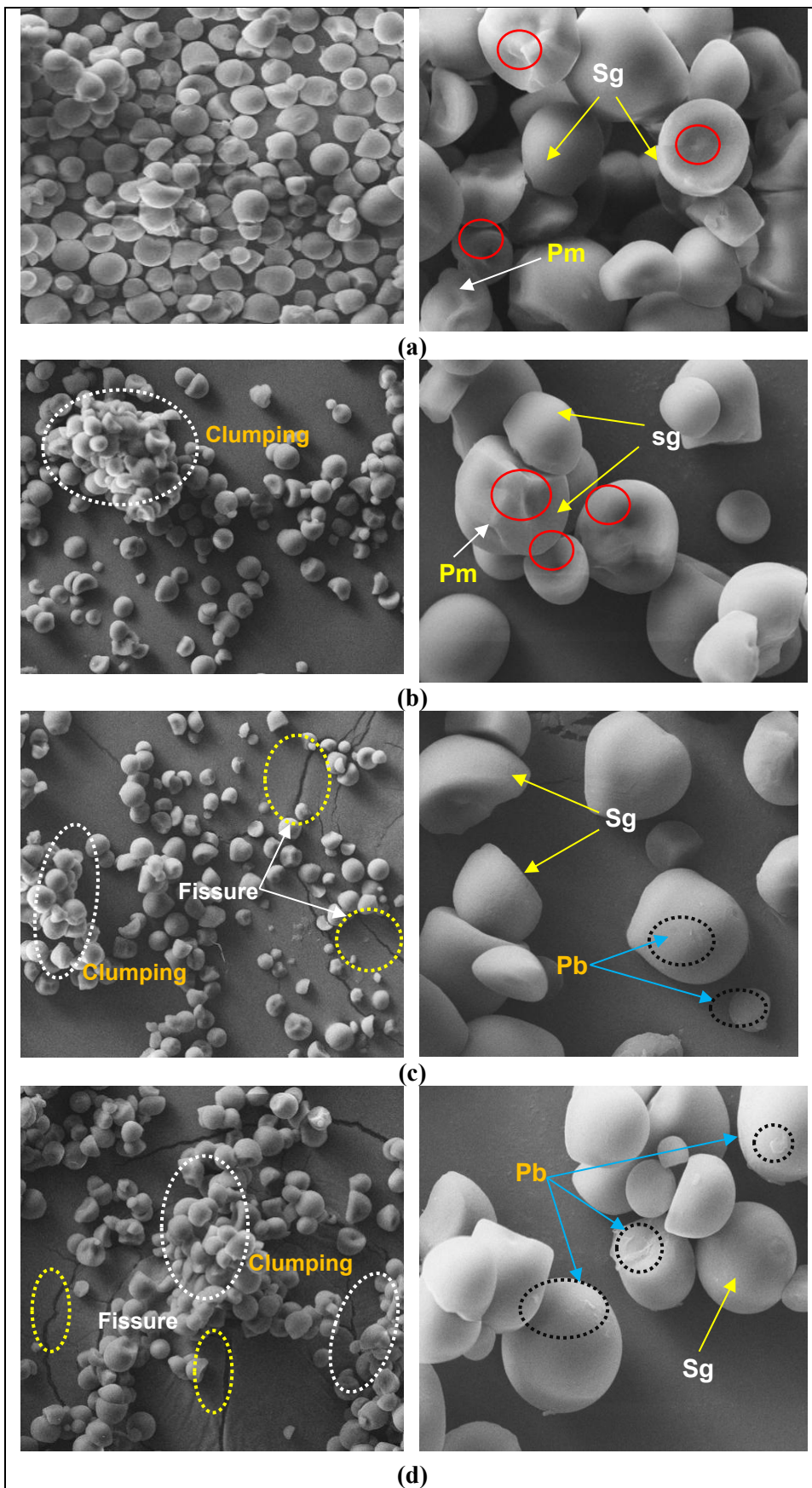


Fig.5. FTIR spectra of native and irradiated cassava starch (Doses: 0, 5, 7.5, 10 kGy)



**Fig.4.** Scanning electron microscope (SEM) images of native and irradiated cassava starch (a) 0 kGy cassava starch, (b) 5 kGy cassava starch (c) 7.5 kGy cassava starch, (d) 10 kGy cassava starch. sg = starch granules, pm = protein matrix, pb = protein bodies.

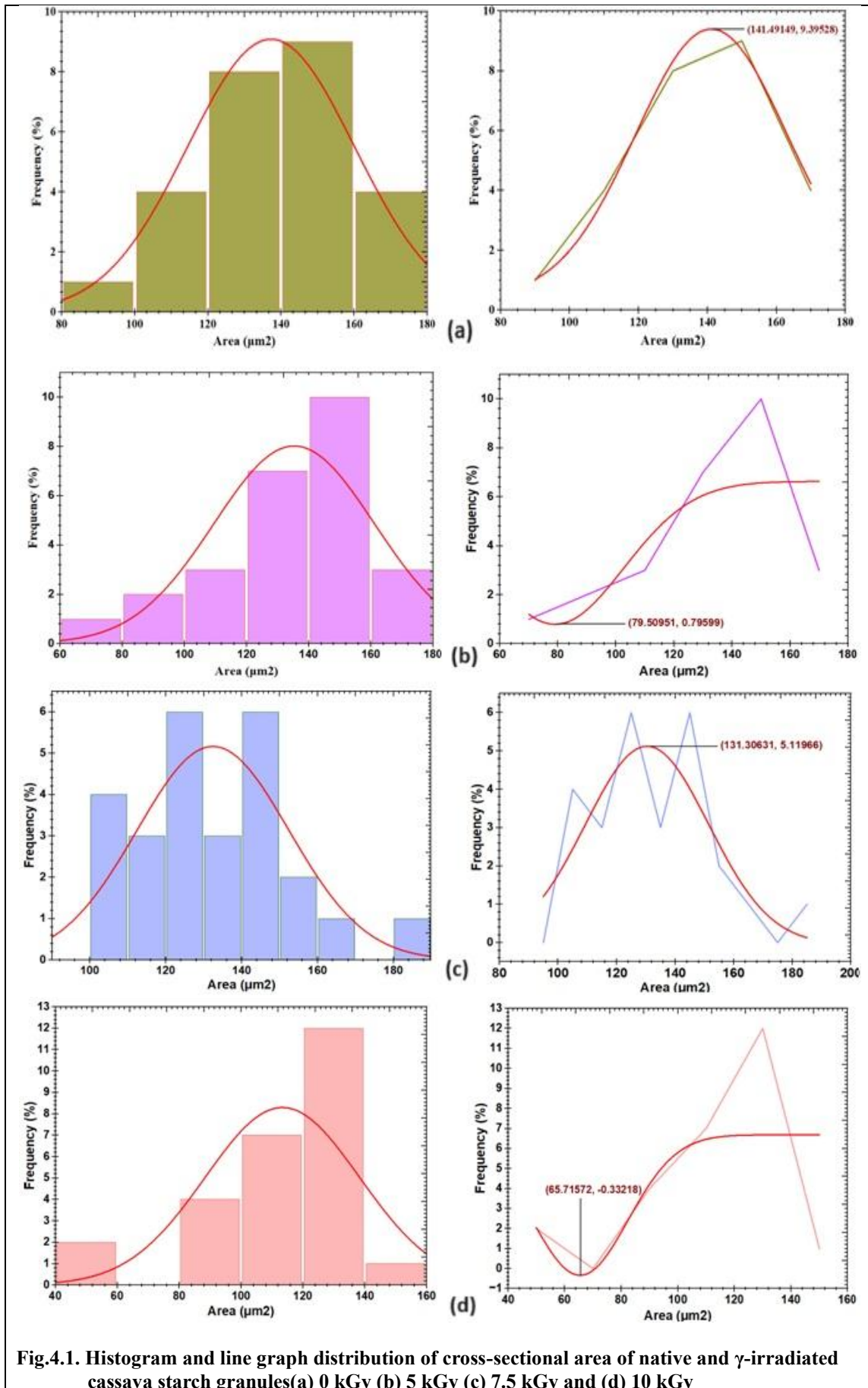
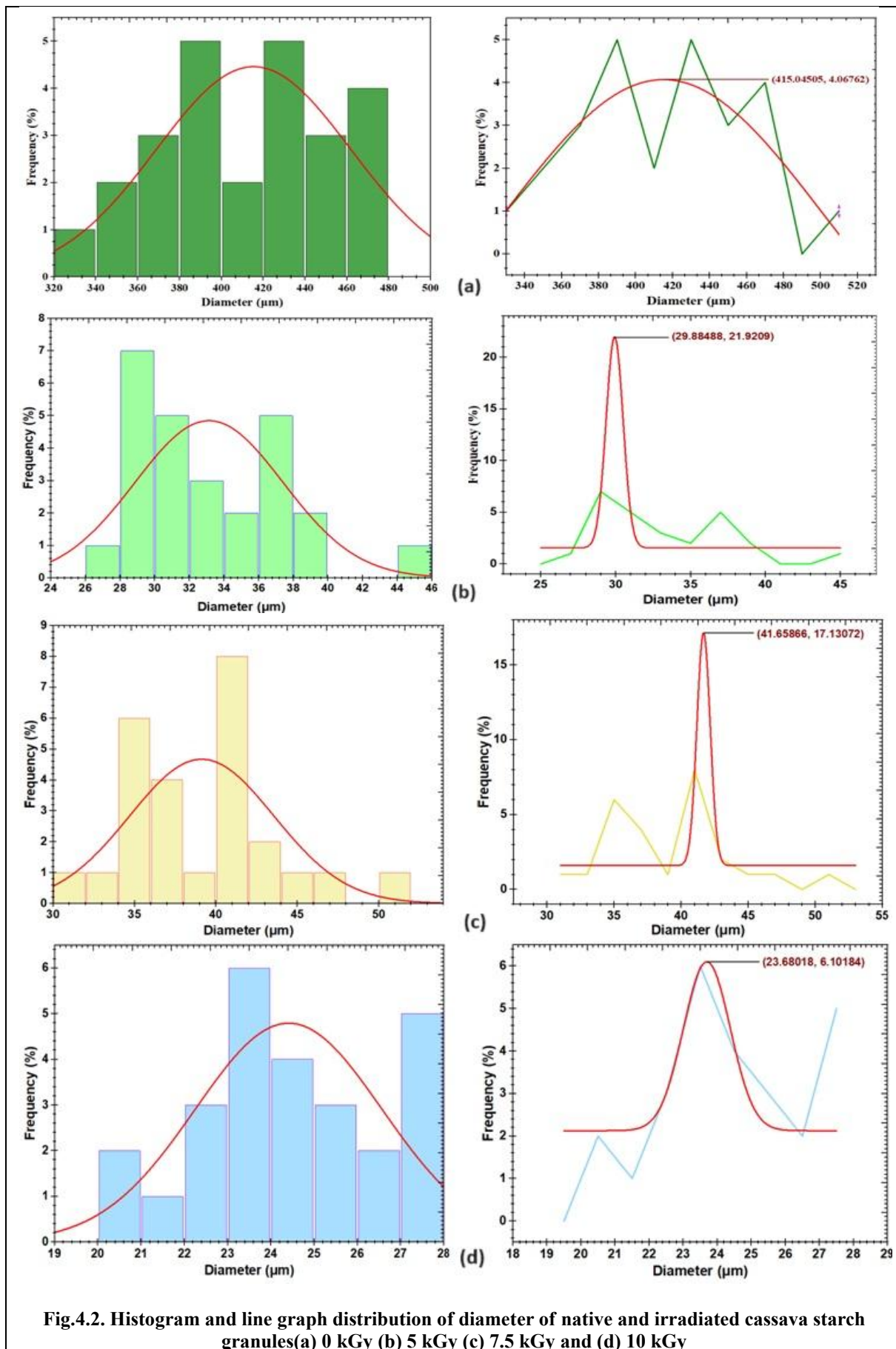


Fig.4.1. Histogram and line graph distribution of cross-sectional area of native and  $\gamma$ -irradiated cassava starch granules(a) 0 kGy (b) 5 kGy (c) 7.5 kGy and (d) 10 kGy



### Conclusion

The  $\gamma$ -irradiated cassava starch product would recommend in future prospect those people suffering from celiac disease in current scenario seen that children are suffer from gluten-intolerance so cassava muffin is a better option to consume it, compare to market muffin. Cassava starch can be used to prepared directly for further processing to develop value added products, edible coating materials, biodegradable packaging films as well as to make a rodent's trappers also. Due to unique properties of cassava starch, it suggests for specialty markets such as baby foods and non-allergenic products.

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### Author's Contribution

Suresh Chandra and Neelash Chauhan guided and designed the experiment and arranged the experimental facilities. Ruchi Verma collected and analyzed the data and also contributed to the writing of the manuscript.

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### Conflicts of interest

The authors have no conflicts of interest to declare. All co-authors have seen and agree with the contents of the manuscript and there is no financial interest to report. We certify that the submission is original work and is not under review at any other publication.

### Data Availability

Physico-chemical properties (%), Swelling and solubility index (g), SEM, and FTIR data are used to support the findings of this study and are included in the manuscript.

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