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Characterization of barley-corn flour based hydrogel and its xerogel formation

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Abstract
Biopolymers like starch and protein have a wide range of applications in the food industry due to their different physicochemical characteristics, such as gelatinization properties. The gelatinization properties of barley and corn create an immense application in developing hydrogel and xerogel. 2-D barley-corn xerogel has a great future in developing 3-D and 4-D snacks. The barley-corn flour mixture with concentrations of 5%, 7%, and 9% with a temperature range of 60 °C, 70 °C, 80 °C, and 90 °C was considered for the xerogel formation. The physicochemical properties were studied during the hydrogel formation. The gelation properties, swelling and soluble nature of the flour mixture, absorbance, and density. The drying characteristics and contact angle were also analyzed during the study. Pasting and rheological properties affect the sessile drop formation and estimating the contact angle. Desired drying conditions are characterized by lower contact angle and lower surface tension of the hydrogel. The higher temperature of hydrogel induces the hydrolysis of starch and the formation of thick turbid consistency, which leads to the formation of cracks during drying. Xerogel formed from barley and corn has a significant effect on nutritional characteristics. The functional group study by FTIR spectrophotometer was also conducted for the further development and application of xerogel.

Keywords: Xerogel, sessile drop drying, hydrogel, pasting properties

1. Introduction
Hydrogels are polymer networks that have been intensively expanded with water. A hydrogel provides both the properties of the liquid (flow properties) as well as solid (elastic properties) (E. M. Ahmed, 2015) [1]. The gels used in the food system have acquired an immense interest in food research, which consists of a significant proportion of aqueous medium (more than 80%). The biopolymeric gels are classified as hydrogel, oleogel, and aerogel, based on the dispersed medium water, oil, and air, respectively. The three-dimensional network biopolymers bonded with water or other dispersed phases provide extensive physical and functional properties (Deligkaris *et al*., 2010) [10]. The biodegradable, edible, non-toxic, bioactive, and sustainable properties of the hydrogel expand the functionality and increase its application in the food industry (Ismail *et al*., 2013) [12]. The gels provide the functions such as thickening agents, retaining food structure, preservative properties, fat mimics or replacers, and improving the sensory properties of the foods (Li *et al*., 2017) [17]. The hydrogels in the biological systems are made from two major polymers: protein and carbohydrate. The carbohydrates consist of guar gum, agar-agar, and starch, whereas protein-based hydrogels are collagen and gelatin-based, zein-based, keratin, and elastin-based hydrogels (Basu *et al*., 2018) [3].

Barley and corn are consumed worldwide as staple cereals; apart from that, it is used to produce breakfast foods, extruded products, noodles, and snacks (Symons & Brennan, 2004) [34]. The barley consists of 65%-72% of starch, whereas the amylose and amylopectin have a ratio of 1:3. Meanwhile, corn flour consists of 62%-67% of starch, while the amylose and amylose are in the same proportion (Lohani & Muthukumarappan, 2017) [20]. The beta-glucan content of barley flour provides health benefits and controls the gelling properties of barley flour suspension. The beta-glucan reduced the gelling properties of barley flour suspension due to the starch-beta glucan formation (Dangi *et al*., 2020a) [8]. The corn starch provides better gelling properties, while the higher sugar content imparts stickiness to the hydrogel (Sun *et al*., 2014) [33].

Xerogel is the solid form of a gel that is formed by drying uniformly without shrinkage. The xerogels are dried using sessile drop drying, also known as evaporative drying, where the hydrogels are dried slowly at room temperature. The various pattern of the sessile drop drying

consists of stick-slip mode (SS), constant contact angle mode (CCA), and constant contact line mode (CCL). The sessile drop is a hemispherical droplet formed when a hydrogel is poured over a non-sticky surface due to surface tension (Choudhury *et al*., 2013) [6]. During the sessile drop drying, the liquids present in droplets evaporate and form solid matter over the contact material (Boopathy *et al*., 2021) [4]. Xerogels retain more surface area due to high porosity after drying the hydrogels (Tüysüz & Schüth, 2012) [36]. Xerogel has different properties consisting of a larger surface area and high porosity, with pore size ranging from 1-10 nm (Gulrez *et al*., 2011) [11]. In addition, the xerogel has morphogenesis properties (top smooth and rough bottom texture), which leads to the shape transformation of the xerogel under different stimuli (Kong *et al*., 2021) [15]. Xerogel has an immense opportunity in the food sector, such as it provides well-engineered and well-structured foods like chips, pasta, and noodles. Moreover, it is used for 3D and 4D printing as well as for the controlled release of nutraceuticals. Fully gelatinized flour produces uniform expansion and more crispiness (Ratish Ramanan *et al*., 2018) [30]. In addition, the anisotropic swelling property of xerogel has provide an extensive opportunities in the food industry due to the different stimuli-responsive shape-shifting properties (Mizuno & Furuya, 2019) [23]. Stimuli like hydrothermal, oil and pH are used to make 2D to 3D and 4D shape transformations.

The type of flour, gelatinization temperature, fiber content, relative humidity, and drying temperature determine the type of xerogel formed and shape transformation (Yang *et al*., 2019) [38]. Different construction materials are used to get the exact shape transformation, and it will determine the shape of the gel. The Retrogradation and recrystallization occurred during the drying of the 3D hydrogel matrix (Jaspin *et al*., 2022) [14]. The study mainly focused on the effect of temperature and concentration upon the formation of barley-corn xerogel and its applications in food. This work dispenses the information regarding the development of barley-corn xerogel, and the possible future applications are discussed in the study.

2. Materials and Methods

2.1 Materials

Barley (*Hordeum vulgare)* and corn (*Zea mays*) flour were procured from Thanjavur market, in Tamilnadu, strained through 106 µm. The flour composition was estimated using the corresponding American Association of Cereal Chemists (AACC) methodologies. The barley-corn flour blend comprises 11.33±0.37 percent crude protein, 2.73±0.21 percent crude fat, 74.65±0.54 percent carbohydrate, 10.72±0.25 percent moisture, and 0.52±0.02 percent ash.

2.2 Least gelation concentration (LGC)

LGC is the lowest concentration where the gel did not slide down from the test tube. LGC was measured using the procedure described by Dangi *et al*., (2020b) [9]. Barley-corn flour suspension was prepared by blending 10 ml deionized water with flour concentrations of 2%, 4%, 6%, 8%, 10%, 12% and 14%. Then, the suspension was gelatinized by heating for 1 hour, then cooled (10 °C) for 2 hours.

2.3 Pasting properties

The properties of barley-corn flour were assessed by a Modular Compact Rheometer (Version; RHEOPLUS, MCR52, Anton Paar, Co. Ltd, Austria). The barley-corn flour suspension was formulated by mixing distilled water (25 ml) and transferred to the canister. The viscoamylograph were obtained for 5%, 7%, and 9% concentrations. The process consists of programmed series of steps, including holding the sample for 50 ℃ for 1 minute, raising the temperature to 95 ℃ for 3.5 minutes (held for 2.5 min), and reducing the temperature to 50 ℃ for 3.8 minutes (then holding for 1.4 minutes). Peak viscosity, trough viscosity, breakdown viscosity, final viscosity, setback viscosity, and pasting temperature were estimated and analyzed from the viscoamylograph (Q. Liu *et al*., 2020) [18].

2.4 Swelling power and solubility

The swelling power and solubility of the barley-corn flour were determined by the procedure explained by Ulfa *et al*., (2020). First, 0.5g of the flour suspension was prepared by adding distilled water (25 ml) and heating up to different temperatures of 60 °C, 70 °C, 80 °C, 90 °C. The flour dispersion was cooled down to room temperature, followed by centrifuged at 3000rpm (15 minutes). Next, the weight of the residue was measured to estimate the swelling power. Finally, dried the supernatant at 105 °C and measured the weight to determine the solubility. The swelling power and solubility are calibrated by the equations (1) and (2).

$$\text{Solubility}(\%) = \frac{A}{S} \times 100 \quad (1)$$

$$\text{Swelling power} = \frac{B \times 100}{S(100 - Solubility\ (\%))} \times 100 \quad (2)$$

Where, A= solid particle weight (g); B = residue weight (g); S = sample weight (g).

2.5 Barley-corn based hydrogel

A suspension of barley-corn flour at various concentrations (5 percent, 7 percent, and 9 percent (w/v)) was produced by heating to temperatures of 60 °C, 70 °C, 80 °C, and 90 °C. The formation of lumps was prevented by constant stirring, and the temperature was maintained throughout the process using a thermocouple. The density, absorbance, angle of contact, and surface tension were measured at each treatment condition.

2.5.1 Density

The hydrogel density at each temperature condition was evaluated by measuring the weight-to-volume ratio of each sample (Tan *et al*., 2019) [35]. The weight of an equal volume of suspension was measured by pouring the sample into the 5 ml graduated cylinder.

2.5.2 Light transmittance

The absorbance of the hydrogel is the function of light transmittance. The light transmittance is determined by measure the absorbance at 640 nm using UV Vis Spectroscopy (Si-can 2301, In-karp instruments Pvt Ltd). The absorbance for barley-corn hydrogel at a temperature of 60 ℃, 70 ℃, 80 ℃, 90 ℃, and 100 ℃ was taken, where water was taken as blank (Kordjazi *et al*., 2020) [16].

2.5.3 Surface tension and contact angle

The barley-corn hydrogel (1ml) was poured over a nonstick (food grade) material surface in front of a GigE vision area

scan camera (C1600, Genie color series, DALSA) attached to a Zoom7000, Navitar. The background light was provided from the back of the droplet to obtain a silhouette image for improved contact angle measurement. The elevation of the droplet was recorded and analyzed by ImageJ software (Fiji - ImageJ 1.52d, National Institutes of Health, USA). For the calibration, the software-plugin (drop analysis-Dropsnake method) with B-spline active contours and Low-Bond Axisymmetric Drop Shape Analysis was used (Qin *et al*., 2021) [27]. Similarly, the hanging droplets from the micropipette were used to analyze the surface tension. When the droplet was detached from the micropipette tip, the image was recorded and analyzed using the plugin Pendant drop (Oropeza *et al*., 2022) [26].

2.6 Xerogel formation

The xerogel was prepared by taking the lower and higher concentrations than LGC (5%, 7%, and 9% (w/v)) was heated to 60 ℃, 70 ℃, 80 ℃, and 90 ℃ to attain the hydrogel. Then, it is transferred (1ml) to a nonstick surface and dried for 10 hours at 29 ℃ and an RH of 76%. The flat 2D structured xerogel (Top smooth and rough bottom morphology) was identified as the optimized xerogel.

2.6.1 FT-IR spectroscopy of barley-corn flour mixture and xerogel

The functional group examined for the barley-corn flour, top side, and bottom side of xerogel was identified using Fourier transform infrared (FTIR) spectrophotometer, PerkinElmer, USA (ATR mode). The band of 4000-400 cm−1 with 16 scans by transmittance mode were used for the analysis (Jafari *et al*., 2017) [13].

2.7 Statistical analysis

The values are recorded as triplicate and evaluated using SPSS (SPSS statistics version 20, IBM). The Duncan multivariable techniques were used to analyze the data; the average difference of the triplicates was evaluated for significance at $p<0.05$. Letters/subscripts were used to denote the significant difference, and the values were represented as the data along with standard error (Mean ± SD)

3. Result and Discussion

3.1 Least gelation concentration (LGC)

The minimum concentration where the gel is retained in the inclined tube is the LGC, which is used as an indicator of the degree of gelation (Mishyna *et al*., 2019) [22]. The LGC of flour depends on the composition of the flour, consisting of amylose and amylopectin content, fiber content, protein content, lipid content, and aging of the flour (Symons & Brennan, 2004) [34]. The higher LGC value denotes the more concentration of flour required to form the gels. The value of LGC obtained for barley-corn flour was 6%. The LGC value for corn flour alone was 6% (Stephen *et al*., 2021) [32], whereas the LGC value for barley flour was also 6% (Onitilo *et al*., 2007) [25]. The lower LGC value denotes better gelling properties of the flour mixture. The LGC varies according to the variety, processing of the flour, its particle size, and the fiber content of the flour (Symons & Brennan, 2004) [34].

3.2 Pasting properties

These pasting profiles are evaluated to confirm the LGC of the flour mixture. The LGC was obtained at 6% concentration, while the pasting properties were determined at different concentrations of 5%, 7%, and 9%, which is above and below the LGC range. The pasting properties are represented in Table 1. The pasting temperature raises with a raise in concentration of the flour**.** The pasting temperature at 5% concentration is 72 ℃, which denotes the higher energy required to swell granules at a lower concentration. The amylose and amylopectin content represent the gelling power and viscosity, respectively (Rafiq *et al*., 2015) [28]. The amylose is leached during heating, leading to the development of a network of hydrogel; this temperature is represented as the pasting temperature. The peak viscosity represents the moisture retention power, while the peak time represents the time taken to obtain the peak viscosity. The gelling power and viscosity values increase rapidly with the change in concentration of the flour mixture. The breakdown, holding, setback viscosity, and final viscosity values increased rapidly with a raise in flour concentration. These pasting properties have a direct relationship with the concentration of barley-corn flour mixture.

Table 1: Pasting profiles for a barley-corn flour blend at 5%, 7%, and 9% concentrations

Parameter	Concentration		
	5%	7%	9%
Pasting temperature (℃)	72.312±0.592^{a}	64.546±0.719^{b}	58.135±1.249^{c}
Peak temperature (℃)	90.661±0.716^{a}	91.296±1.842^{a,b}	92.352±0.228^{b}
Peak Viscosity (cP)	17.924±0.247^{a}	76.468±3.146^{b}	162.425±5.467^{c}
Holding viscosity (cP)	5.675±0.495^{a}	42.546±8.107^{b}	84.567±4.765^{c}
Break down viscosity (cP)	8.865±0.452^{a}	35.965±2.624^{b}	62.354±2.154^{c}
Final viscosity (cP)	52.964±0.214^{a}	485.468±22.657^{b}	627.497±27.657^{c}
Setback viscosity from the peak (cP)	-41.254±0.647^{a}	-384.457±11.456^{b}	-496.627±38.954^{c}
Setback viscosity from the trough (cP)	54.954±2.645^{a}	249.256±24.459^{b}	543.354±35.354^{c}

3.3 Swelling power and solubility

The solubility and swelling power were determined to study the impact of temperature on the gelling properties of the barley-corn hydrogel. The swelling and solubility values with respect to the temperature have given in Table.2. A gradual raise in the solubility, and swelling power values were observed along with temperature increase. The solubility increased rapidly from 70 ℃ to 80 ℃ due to the higher energy and dissociation of amylose and amylopectin. The gelatinization of barley flour starts at 57 ℃, and the complete gelatinization occurs at 75 ℃ (Neill *et al*., 2012) [24], whereas the gelatinization of corn occurs at 70 ℃ to 75 ℃ (Coral *et al*., 2009) [7]. This rapid hike in gelation is due to the higher gelatinization temperature of barley and corn flour. When the suspension reaches the temperature above the gelatinization temperatures, amylose leaches out, and the crystalline nature of starch molecules is reduced and attains more amorphous properties, which leads to more swelling and solubility of the

suspension.

3.4 Hydrogel formation

3.4.1 Density

The density was determined to study the impact of temperature on the gelation properties, illustrated in Table.2. The density of the suspension increased from 60 ℃ to 90 ℃ due to the reduction in crystallinity and improved solubility of the starch. The higher temperature of starch leads to increases in the amorphous property, which improves the solubility and hydrophilicity of the suspension (Boopathy *et al*., 2021) [4]. This change causes the breakdown of more intermolecular and intramolecular hydrogen bonds, which are linked with more hydroxyl groups and improve swelling of starch. The high turbidity and viscosity at higher temperatures are an indication of hydrogel formation. The hydrophobic nature of the suspension at a lower temperature provides a lower density, while the gelatinized (swollen) particle at a higher temperature provides a higher density.

Table 2: Physical characteristics of barley-corn hydrogel at varying temperatures

Parameter	Temperature			
	60 ℃	**70 ℃**	**80 ℃**	**90 ℃**
Solubility (%)	2.796±0.047^{a}	3.215±0.197^{b}	7.631±0.465^{c}	9.632±0.743^{d}
Swelling power	3.972±0.357^{a}	5.137±0.307^{b}	6.725±0.159^{c}	8.713±0.249^{d}
Absorbance	2.873±0.047^{a}	2.427±0.197^{b}	2.173±0.465^{c}	1.975±0.743^{d}
Contact angle (°)	62.248±0.987^{a}	65.752±0.349^{b}	67.441±0.007^{c}	69.190±0.048^{d}
Density (kg/m^{3})	0.884±0.047^{a}	0.902±0.197^{b}	0.957±0.065^{c}	1.002±0.043^{d}
Surface tension (mN/m)	45.153±0.762^{a}	52.062±1.543^{b}	57.149±2.269^{c}	68.648±0.350^{d}

3.4.2 Light transmittance

The light transmittance was determined at 640nm by a UV-Vis spectroscopy. The absorbance of the suspension has an inverse correlation with the gelling power of the flour suspension. Light transmittance is the function of absorbance, where the absorbance value is reduced with an increase in temperature while light transmittance increases. The change in absorbance value with temperature is represented in Table 2. After the complete gelatinization, the crystallinity and birefringence get reduced along with the leaching out of amylose (Martins *et al*., 2019) [21]. The rate of scattering inside the hydrogel was decreased due to the loss of crystallinity and improved light transmittance. The complete gelatinized starch possesses a highly ordered structure due to its amorphous nature, which leads to better transmittance and lower absorbance.

3.4.3 Surface tension and contact angle

The contact angle is measured by taking the side view (silhouette image) of the sessile drop, which is the angle between the flat surface and the tangent of the semispherical surface of the sessile drop. The contact angle depends upon the polarity of the droplet as well as the property of the surface. The sessile drop was formed after pouring the hydrogel over a nonstick surface; with temperature rises, the contact angle increased substantially. The increase in contact angle is due to the cohesion properties of the hydrogel, while the highest contact angle is obtained at 90 ℃. In addition, the hydrophilicity of the hydrogel was increased with an increase in temperature due to more solubility and swelling power (W. Liu *et al*., 2004) [19].

The surface tension of the hydrogel was determined by pendent drop mode, which was found to be a suitable technique (Stephen *et al*., 2021) [32]. The surface tension is the critical parameter, in determining the drying characteristics (M. A. Ahmed *et al*., 2016) [2]. The extent of gelatinization increases the surface energy. In addition, the cross-linking of the polymers occurred during gelatinization at a higher temperature, which reduces the suspended particles in the hydrogel. The gelatinization and cross-linking increase the surface energy, which leads to the formation of more spherical droplets and, thus, creates higher surface tension.

3.5 Effect of temperature and concentration on the xerogel formation

The barley-corn xerogel was formed by the sessile drop drying at 29 ℃ and an RH of 76% for 10 hours. Uniform drying occurred throughout the process. The barley-corn hydrogel with 5%, 7%, and 9% concentrations with 60 ℃, 70 ℃, 80 ℃, and 90 ℃ was used to produce the xerogel by sessile drop drying as illustrated in Figure.1. The flat xerogel was obtained at 5% (80 ℃) and 7% (70 ℃) concentration. At the same time, the depinning is difficult at 5% concentration due to the thinner cross section compared to the 7% concentration. At the same time, the desired xerogel was obtained at a 7% concentration and a temperature of 70 ℃. The proper hydrogel was formed at 70 ℃ to 90 ℃, while at 60 ℃, the cracking was observed due to the improper gelatinization and suspension of particles. The standard modes of sessile drop drying are constant CCA, CCL, and mixed mode, while barley-corn flour follows CCL. The fragmentation of starch granules occurs at higher temperatures, which leads to the marangoni effect and coffee staining effect (Carreón *et al*., 2021) [5]. This effect leads to the precipitation of the fragmented particles towards the periphery (Sefiane, 2014) [31]. At 90 °C, a hydrogel with 5%, 7%, and 9% concentrations shows a coffee staining effect due to the fragmentation of starch granules precipitated towards the periphery. More minor cracks were observed at 9% concentration and 90 ℃, due to the capillary flow pattern and marangoni effect.

The hydrogel at a lower temperature (60 ℃) contains partially gelatinized starch suspended during the drying process. The suspension of the particles leads to cracks and the powdery nature of the xerogel. The lower concentration enhances the formation of flat xerogel due to the desired viscosity and surface tension of the sessile drop. In comparison, the higher concentration of hydrogel shows the marangoni effect. The flat xerogel was formed at a lower temperature above the gelatinization temperature, while at a higher temperature, the starch granules were fragmented, which leads to the coffee stain effect, and causes the bending or curling of the xerogels.

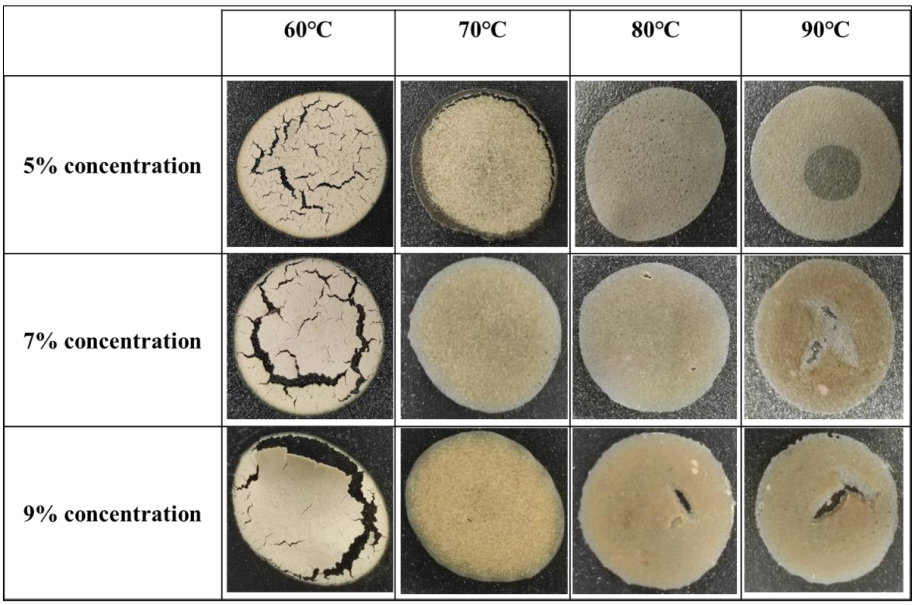

Fig 1: Effect of temperature and concentration on barley-corn xerogel formation

3.5.1 FT-IR spectroscopy of barley-corn flour mixture and xerogel

The changes in functional group of the xerogel and flour during the gelatinization and drying processes were analyzed and characterized by FTIR spectroscopy has illustrated in Figure 2. The gelatinization and retrogradation process that changes in the hydroxyl as well as other functional groups (Jafari *et al*., 2017) [13]. The movement of the swollen and suspended particles during the sessile drop drying creates a gradient in the functional group present in the top and bottom of the xerogel (Ramanan & Mahendran, 2021) [29]. 3300 cm^{-1} represents the -OH group, and there is a significant change in the -OH group due to gelatinization and retrogradation. The crystallinity or amorphous nature of xerogel was determined by the band at 1000 cm^{-1}. At the same time, the structural characterization is determined by the region ranges from 1350 cm^{-1} to 930 cm^{-1} (Boopathy *et al*., 2021) [4]. There is no characteristic variation in the overall functional group of xerogel and barley-corn flour mixture.

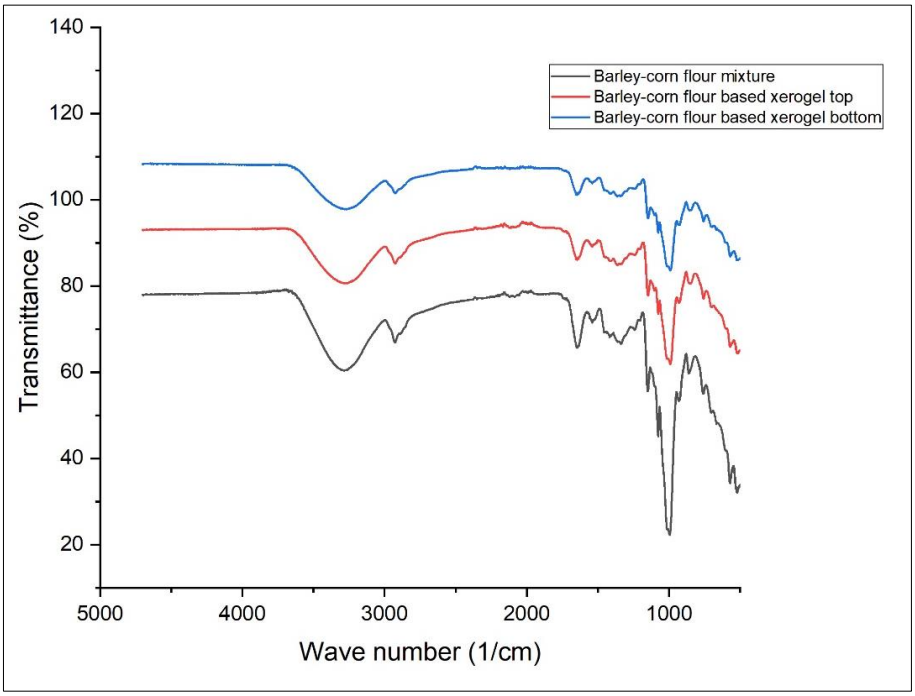

Fig 2: FTIR spectra of barley-corn flour, and top and bottom sides of barley-corn flour based xerogel

4. Conclusion

The impact of the concentration as well as the temperature on the formation of barley-corn xerogel formation was studied. The study highlighted the effect of the temperature and concentration of the barley-corn flour on the physicochemical parameter and its impact on the xerogel formation. The flat suitable barley-corn xerogel was obtained at a concentration of 7% and 70 °C temperature. The xerogel was not formed at lower concentration due to improper gelatinization. At the same time, at a higher temperature, it shows an irregular

shape due to the precipitation of particles towards the periphery. The crack-free barley-corn xerogel has a potential application in the food processing sector in terms of nutraceutical development, 3D and 4D shaped transformation, and functional food development. The porous structure provides better adsorption property to the xerogel. Moreover, the biodegradable property of xerogel makes it a suitable carrier for bioactive components. The carrier properties, bioavailability, and stability after the development of functional foods are yet to be discovered.

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