# Refractance Window Drying Of Aonla Pulp To Produce Powder

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**Abstract**
Aonla fruits are found to be rich in ascorbic acid and have been reported to be useful in curing many diseases. Refractance Window (RW) drying is a novel thin-film drying process, generally used for drying of heat sensitive farm produce for retention of necessary quality attributes. Aonla pulp of 3 mm thickness was dried using RW drying process to obtain powder. RW drying was conducted to study the single layer drying kinetics, and other quality parameters such as colour, thermal properties, sieve analysis, FT-IR and SEM analysis. RW drying of 3 mm thick pulp entered into falling rate drying period after 6 min of constant rate drying. The diffusivity of moisture values for 3 mm thick pulp RW drying was 5.48 x $10^{-10}$ to 3.5 x $10^{-9}$ $m^2$ $s^{-1}$. The dried powder was bright, more yellow and less green as compared to pulp. FT-IR results show that shift of spectrum peaks were more pronounced for 212 µm powders particles than for 500 µm powder particles with respect of pulp. Ascorbic acid presence is characterized by peaks obtained at 1633 cm-$^{-1}$, 1741 $cm^{-1}$ in IR spectrum of pulp, 1747 $cm^{-1}$ and 1629 $cm^{-1}$, 1749 $cm^{-1}$ in 500 µm and 212 µm powders particles, respectively. SEM analysis for powder particles shows that the RW dried powder particles were of uniform thickness and smooth surfaces.

**Keywords:** Aonla powder, Refractance Window drying, thermal properties, sieve analysis, FT-IR analysis

## 1. Introduction

Anola, also well-known as Indian Gooseberry (*Emblica officinalis* Gaertn.), has got tremendous medicinal value for that reason it has a distinctive place in India (Chandane 2015). The fruits are rich in vitamin C and tannins. Anola friuit vitamin C content was found to be 30 times higher than oranges (Annapurna 2012). Anola fruits have been reported to be useful in curing anaemia, haemorrhages, dysentery, diarrhoea, jaundice, cough and dyspepsia. High fibre content of anola fruit makes it helpful in easing constipation and regulating bowl movements (Annapurna 2012). Anola is used to prepare well-known indigenous medicines in Ayurvedic system such as chavanprash and trifla (Chandane 2015). India produced 1073.69 thousand tones Aonla in 2017-18 (Anonymous 2022).

The shelf life of anola fruit at ambient condition varies from 8 to 10 days because of its highly perishable nature (Singh et al. 2005). For increasing the shelf life of fruits, drying and dehydration are the preferred processes from ancient time, which eases the cost of transportation, storage, handling and packaging (Kumari and Khatkar 2018). The deterioration reactions and microbial growth are minimized as a result of the reduction of moisture content to the level less than 9%. Drying of fruits and vegetables results in alterations of nutritional and physico-chemical quality attributes like texture, flavour and colour (Di Scala and Crapiste 2008). Formerly sun drying was practiced for anola drying or dehydration and nowadays other drying processes are practiced such as tray drying (Lavanya et al. 2016), cabinet drying (Chandane 2015; Kumari and Khatkar 2018), hot air oven drying (Prajapati et al. 2011), etc.

Refractance window (RW) drying process is a fourth generation dehydration method which was founded by MCD Technologies (Tacoma, Washington, USA). This drying process is developed to produce fruit powders with the advantage of necessary retention of colour, flavour and nutrients (Magoon 1986). RW drying process and equipment were invented for drying and dehydration of thin film fruit pulp or puree and similar kind of materials (Magoon 1986; Nindo 2008).
Literature availability is limited on RW drying of aonla fruit. Therefore, work was planned to study the effect of refractance window drying on aonla fruit pulp, drying kinetics of pulp during RW drying and evaluate different physico chemical properties of dried powder as well as to study the SEM analysis of RW dried aonla powder.

## 2. Material and methods

### 2.1 Pulp Extraction

One kg aonla fruit was procured form Technology market of IIT Kharagpur. Aonla fruits were washed, cut into half, cooked with hot water to remove astringent and bitter taste of aonla (Chandane 2015). Cooled and cooked aonla was

ground to make pulp and preservative 0.75 % Potassium meta-bisulphite was added to it before storage or drying (Shende and Datta 2019). Pulp was stored for determination of quality attributes and for further drying process. Quality attributes of aonla pulp and powder such as colour, water activity, thermal properties, sieve, FTIR and scanning electron micrography (SEM) analysis were carried out.

**2.2 Refractance window drying**

A float prepared from Mylar sheet (DuPont) having dimensions as 24 × 9 × 3 cm, was used to carry out experiments in batch mode (Shende and Datta 2019). Thin and uniform film of aonla pulp 3 mm thickness was spread manually on the top sheet of Mylar float. Refractance window drying was performed (batch wise) at 95 ± 2 °C temperature in hot water bath (S. D. Instruments & Equipments, Kolkata, India) as shown in Fig. 1 (Nindo et al. 2003; Zotarelli et al. 2015). The sample in Mylar float was put on the heated water surface in water bath. The temperature of sample while RW drying was maintained > 75 °C for almost all the time, as indicated by Type-J thermocouple (Shende and Datta 2019).

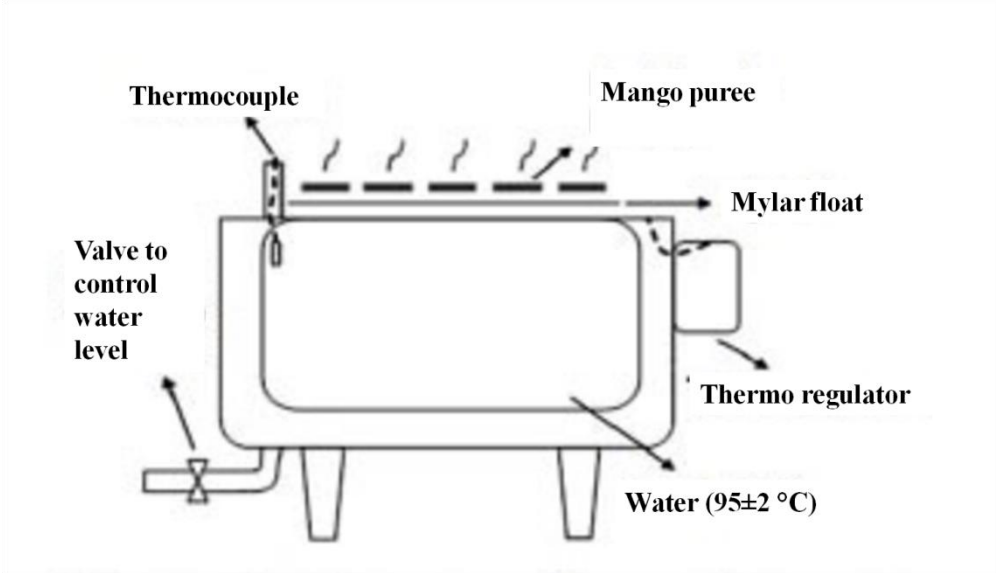


**Fig. 1:** Refractance Window drying of anola pulp using thermostatic water bath (Shende and Datta 2019)

**2.2.1 Single layer drying kinetics of Aonla pulp**

The total weight of float assembly was measured at 2 min drying intervals using digital balance (Sartorius) while RW drying of aonla pulp (Shende et al. 2019). The thickness of the dried aonla flakes was measured using dial guage (Mitutoyo 7301 Dial Indicators). The residual moisture in the RW dried aonla powder was determined using Hot air oven (Orion, India). The required moisture content for fruits and vegetables powder should be in the range of 2 to 5 % (wb) (Caparino et al. 2012; Shende and Datta 2019). The drying kinetics and quality characteristics of aonla powder such as colour, water activity, thermal properties, sieve analysis and scanning electron micrography (SEM) analysis were examined using standard procedures as described below:

**2.2.2 Colour**

Chromameter (CR- 400, Konica Minolta Inc., Japan) was used to measure the colour parameters of aonla pulp and powder (Shende et al. 2016). The parameters of colour for aonla powder such as lightness (L*), red-green (a*), yellow-blue (b*) and total colour difference (ΔE) evaluated were and compared with aonla pulp (Tontul and Topuz 2017; Shende et al. 2019).

**2.2.3 Water activity**

Water activity of aonla pulp and powder were evaluated using digital water activity meter (CX-2 Aqua Lab) (Ochoa-Martínez et al. 2012; Shende et al. 2020).

**2.2.4 Thermal Properties**

Thermal properties of aonla pulp and powder such as thermal diffusivity (D), specific heat capacity (C), thermal conductivity (k), thermal resistivity (ρ) and temperature (T) were determined using KD2 Pro, thermal properties analyzer (Decagon Devices, Inc., USA) (Sharma et al. 2013; SOUSA et al. 2016).

**2.2.5 Sieve Analysis**

RW dried aonla powder was sieved using a series of seven sieves of various apertures and separated into classes using GEOSYN sieve shaker (Test Seive ISS 460, Geologists' Syndicate (Pvt.) Limited, Kolkata). Powder was placed on upper sieve and lower sieves were installed on operating by vertical vibration at 0.5 mm vibration amplitude. Sieving was performed in 3 batches of 10 g, for 10 min each (Sharma et al. 2013). The retained aonla powder on each sieve was

collected and weighed (Sahin and Sumnu 2006). Then, powder samples were packed in zip lock polyethylene bags and stored in refrigerator at 8-10 °C for further quality parameter analysis.

**2.2.6 FTIR analysis**

The FT-IR analysis of aonla samples was done for both in the form of powder and pulp. For analysing aonla pulp samples, a liquid cell is used to be placed in the IR beam in FT-IR Spectrometer (M/S Thermo Fisher Scientific Instruments, 5225 Verona Road, USA) for analysis. Aonla powder sample was ground intimately with mortar-pestle with solid KBr and mixture was converted into a thin pellet using a hydraulic press die (Coates 2000; Zhao et al. 2015). These thin pellets can be put inside the IR beam chamber for FT-IR analysis.

**2.2.7 Scanning Electron Micrography (SEM)**

Aonla powders from RW drying process (212 µm) was spread on aluminium stub in small amount and samples were gold coated using Sputter Coater (POLARON-SC7620, Model-CA76, UK) (Caparino et al. 2012; Shende and Datta 2019). Scanning Electron Micrography of powdered samples were done at 20 kV accelerating voltage using Scanning Electron Microscope (Carl ZEISS SMT, EVO 60, with Oxford EDS Detector, Germany) (Caparino et al. 2013; Shende and Datta 2019). The magnifications of 500 X and 1.0 K X were selected for Micrographs to be photographed at scale bar of 20 µm and 10 µm.

**3. Results and discussion**

Aonla pulp was dried using RW drying to make powder. Pulp layer of 3 mm thickness was spread over float which shrank and became 1.15 ±0.01mm after RW drying. It took 36 min to reach moisture content from 81.52 % (wb) of pulp (Fig.2 (a)) to 4.36 % (wb) powder (Fig. 2(b)).

**Fig. 2(a):** Uniformly spread Aonla fruit pulp over Mylar sheet for RW drying

**Fig. 2(b):** Aonla fruit powder (4.36 % (wb)) after RW drying

**3.1 Single layer drying kinetics of Anola pulp**

The drying of single layer of 3 mm thick aonla pulp took 36 min to get constant mass and got dried from 81.52 % (wb) of aonla pulp to 4.36 % (wb) powder (Fig.2(b)). Initially, the aonla pulp moisture content was observed as 4.24 kg water/ kg dry weight and as a result of RW drying for 36 min final moisture content of flakes layer was measured as 0.0456 kg water/ kg dry weight (4.36%, wb) (Fig. 3(a)). It was observed that, for initial 6 min of drying time the constant rate drying of single layered pulp was occurred. Average drying rate in constant rate of drying was determined to be 0.0091 kg water $m^{-2}$ $s^{-1}$ (Fig. 3(b)). Afterwards, the further drying of aonla pulp was observed to be entered into the falling rate region with 0.0041 kg water $m^{-2}$ $s^{-1}$ as average drying rate.

Moisture diffusivity is considered as important mechanisms while the study falling rate period drying (Shende et al. 2019). From the plot of natural logarithm of moisture ratio (ln X/Xc) versus drying time (Fig.3(c)), the straight line slope was

used to calculate the value of effective diffusivity. The range of diffusivity values was 5.48 x $10^{-10}$ to 3.5 x $10^{-9}$ $m^2$ $s^{-1}$ (Fig. 3(d)).

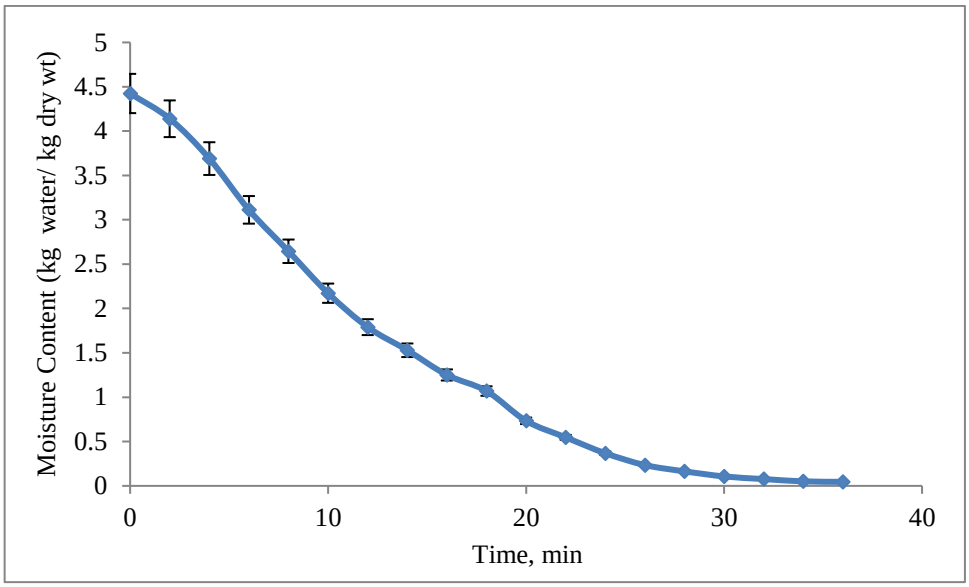


**Fig. 3(a):** Variation of moisture content (d. b.) and time (min)

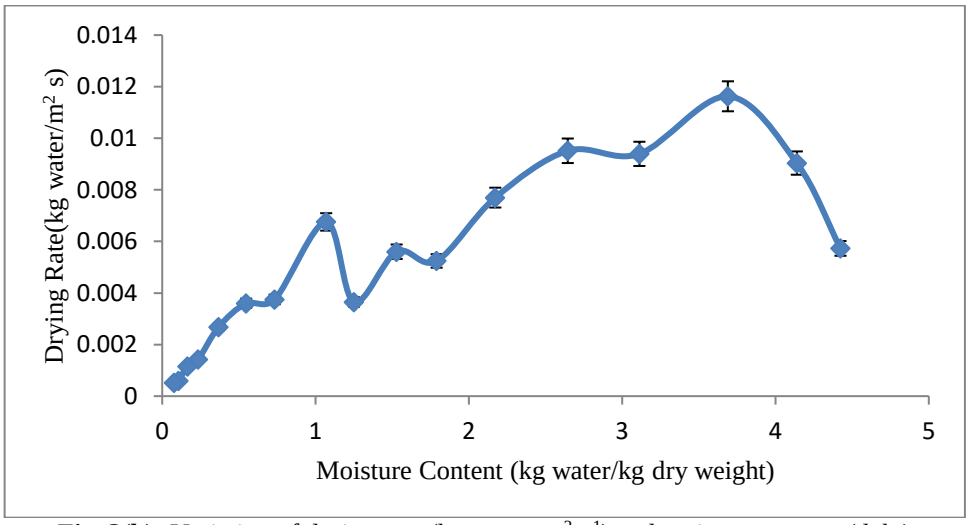


**Fig. 3(b):** Variation of drying rate (kg water $m^{-2}$ $s^{-1}$) and moisture content (d. b.)

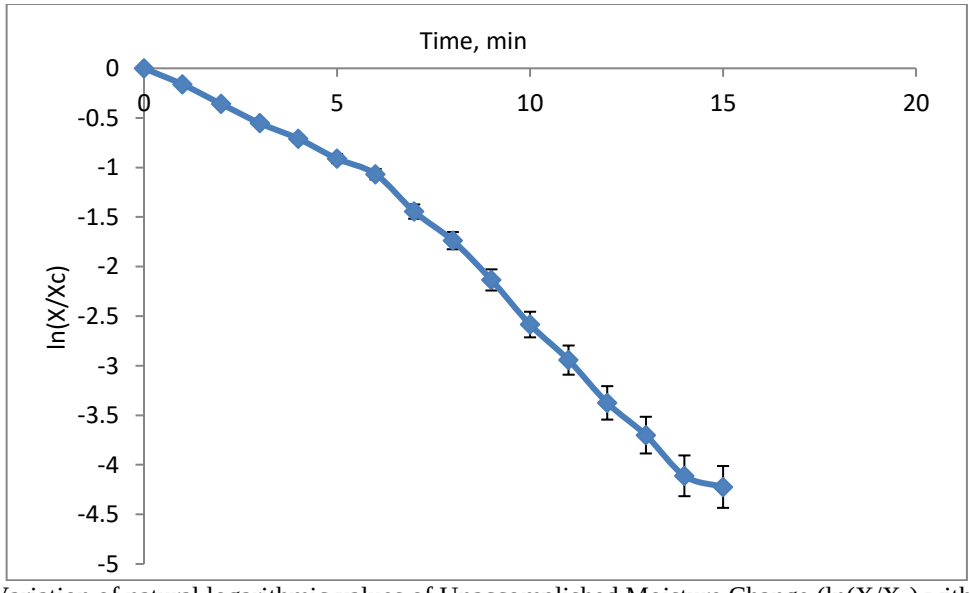


**Fig. 3(c):** Variation of natural logarithmic values of Unaccomplished Moisture Change ($ln(X/X_c)$ with time (min)

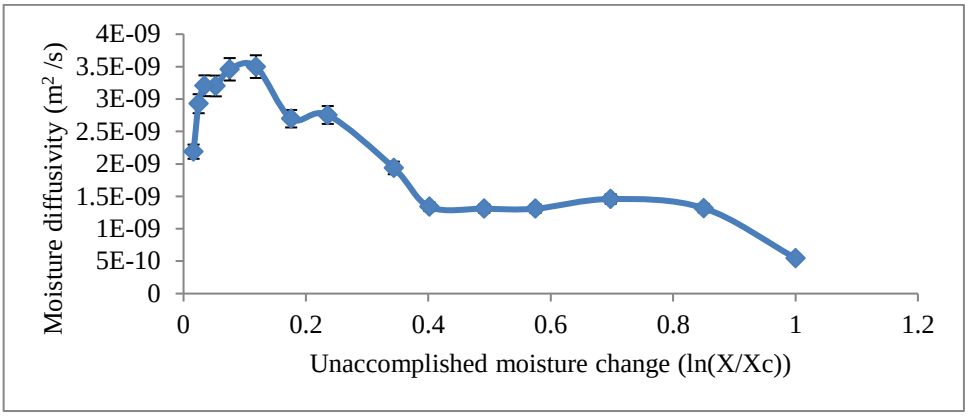


**Fig. 3(d):** Moisture diffusivity curve

### 3.2 Colour

The L* value of aonla pulp was 60.75±0.50 and for powder the same was 70.36±0.35, which shows that after drying the colour of powder becomes more bright as compared to pulp. The a* value of aonla pulp was -1.37±0.05 and for powder the same was 3.34±0.19, which shows that the powder was less green and moved towards red colour of a* value as compared to pulp colour. And the b* value of the aonla pulp was 12.73±0.12 and for powder the same was 30.37±0.28, which shows the powder was more yellow as compared to pulp colour. The total colour difference (ΔE) value was 20.63 for powder colour compared to pulp, which shows that RW drying retains the colour of drying material which was comparable with freeze drying process (Caparino et al. 2012) and better as compared to other conventional drying methods for example hot air drying, oven drying (Shende and Datta 2019), drum drying (Clarke 2004) etc.

### 3.3 Water activity ($a_w$)

The water activity value of aonla pulp was 0.889±0.01, whereas for powder it was measured as 0.352±0.00. After RW drying, the water activity of the pulp was reduced because of depleting moisture within pores or capillaries in the sample (Yoha et al. 2019). Good product stability of powder as compared to pulp was emphasized by Pavan et al. 2012. Aonla powder water activity ($a_w$) after RW drying fall in the range <0.40 where yeast, mold and other microorganism growth were suppressed to extreme low counts (Huang and Hsieh 2005). The dried products can be stored for more than 6 months at ambient temperature and environment with water activity ($a_w$) value <0.50 because of lower $a_w$ is harsh on any microorganisms seeking growth (Huang and Hsieh 2005).

### 3.4 Thermal properties

The thermo-physical properties are very important for the pulp being favored in an industrial level (SOUSA et al. 2016). Thermal properties of pulp and dried aonla powder were determined and it was observed that with the decrease in moisture content, thermal diffusivity (D), specific heat capacity (C) and thermal conductivity (k) values decrease as shown in Table 1. As for aonla pulp thermal diffusivity (D) value of 0.217±0.01 $mm^2\ s^{-1}$ decreased after RW drying to 0.168±0.01 $mm^2\ s^{-1}$ because of lower moisture content of powder. Similarly, the specific heat capacity (C) and thermal conductivity (k) values for pulp were 2.171±0.12 mJ $m^{-3}\ K^{-1}$ , 0.386±0.01 W $m^{-1}K^{-1}$ respectively, which decreased with reduced moisture content and its value for aonla powder after RW drying was 0.631±0.02 mJ $m^{-3}K^{-1}$ and 0.132±0.01 W $m^{-1}K^{-1}$ respectively. Muramatsu et al. (2010) observed that thermal conductivity of orange and grape juice decreases with the increase of concentration of juices from 10 to 50 °Brix.

However, thermal resistivity (ρ) value was 238.66±28.92 °C cm $W^{-1}$ for aonla pulp and 768.86±23.00 °C cm $W^{-1}$ for powder, this increase in value of thermal resistivity (ρ) may be due to the contrary changes in values for thermal conductivity (k) (Sharma et al. 2013).

**Table 1: Quality parameters of aonla powder**

| **Quality Parameter** | **Pulp** | **RW dried Aonla Flakes** |
|---|---|---|
| Drying Time (min) | - | 36±0.5 |
| Thickness (mm) | 3.00 | 1.15±0.01 |
| Moisture Content (%w.b.) | 81.87±0.21 | 4.36±0.02 |
| L* | 60.75±0.50 | 70.36±0.35 |
| a* | -1.37±0.05 | 3.34±0.19 |
| b* | 12.73±0.12 | 30.37±0.28 |
| ΔE | - | 20.63 |
| $a_w$ | 0.889±0.01 | 0.352±0.00 |
| **Thermal Properties** | | |
| Thermal diffusivity (D), $mm^2\ s^{-1}$ | 0.217±0.01 | 0.168±0.01 |
| Specific heat capacity (C), mJ $m^{-3}K^{-1}$ | 2.171±0.12 | 0.631±0.02 |
| Thermal conductivity (k), W $m^{-1}K^{-1}$ | 0.386±0.01 | 0.132±0.01 |
| Thermal resistivity (ρ), °C cm $W^{-1}$ | 238.66±28.92 | 768.86±23.00 |
| Temperature (T), °C | 31.29±0.21 | 27.343±0.50 |

### 3.5 Sieve Analysis

Aonla pulp was dried using RW drying process and after 36 min flakes of uniform thickness were obtained. Fine grinding of aonla flakes was done for size distribution analysis of powder, as shown in Table 2, which shows that more than >60% of the powder sample fall in the range of 355-212 μm size. Also, 12.51 and 12.7 % of powder samples were retained by sieve size >150 μm and >355 μm respectively. For 500 μm sieve size, 9.16 % samples were retained, whereas oversize (>710 μm) particles retained were 2.47 % and undersize (<150 μm) particles retained were 2.30 % of powder sample. Sharma et al. 2013 has observed that with the change in particle size, the physico-chemical properties of the dried sample such as colour, thermal properties, rehydration ratio and sensory properties of reconstituted juice change. It was observed that with the decrease in particles size the lightness of the samples increases and 'a' value decreases linearly indicating the decrease in redness of the samples. The effect of particle size of mango powder was also observed to have impact on thermal conductivity and resistivity and heat transfer in between powder particles (France and Choi 2007; Sharma et al. 2013). This may be due to the fact that with the decrease in particle size, the increase in interfacial area of contact was observed between the powder particles which make the heat transfer process faster (France and Choi 2007).

**Table 2: Sieve analysis of aonla powder**

| Sieve Size (micron) | Weight (g) | Weight Percentage (%) |
|---|---|---|
| 850 | 0.089±0.002 | 0.88 |
| 710 | 0.157±0.002 | 1.59 |
| 500 | 0.916±0.001 | 9.16 |
| 355 | 1.271±0.001 | 12.70 |
| 212 | 6.084±0.001 | 60.85 |
| 150 | 1.250±0.001 | 12.51 |
| Tray | 0.230±0.001 | 2.30 |
| **Total** | **10.000** | **100.00** |

### 3.6 FTIR analysis

The IR spectrum of aonla pulp and RW dried aonla powders (212 and 500 μm) are shown in Fig. 4. The FT-IR spectrum exhibits the characteristic fingerprint band features. The absorption bands and respective assignments are presented in Table 3. The FTIR spectroscopy was determined to identify various components and to evaluate the effects of grinding with respect to pulp. The results show that grounded aonla powders (212 and 500 μm) did not differ in chemical groups with respect to the pulp. But the shift of peaks was observed while small variation was found in peak intensities. Shift of peaks was more pronounced in 212 μm powder particles than for 500 μm powders particles with respect to the pulp. This may postulate that the chemical functional groups of powders were directly related to variations in granular size of the powders. This attributed to swapping of the surface properties of powders having different fineness. Whereas, change in peak intensities followed opposite trend than shift of peaks (Fig. 4). The absorption band peaked at 3446 $cm^{-1}$ for pulp, 3457 $cm^{-1}$ and 3428 $cm^{-1}$ for 500 μm and 212 μm powder particles respectively, indicating O–H stretching of hydrogen-bonded hydroxyl groups. The presence of ascorbic acid is characterized by peaks obtained at 1633 $cm^{-1}$, 1741 $cm^{-1}$ in IR spectrum of pulp, 1747 $cm^{-1}$ and 1629 $cm^{-1}$, 1749 $cm^{-1}$ in 500 μm and 212 μm powders particles, respectively. These peaks are representing presence of ester group and C=O stretching vibration (Pantwalawalkar et al. 2020). The peaks obtained in the range of 2944 $cm^{-1}$ to 2857 $cm^{-1}$ show that powder obtained at 212 μm powders particles shifted to lower wavenumber (i.e. 2857 cm-1) as compared to pulp. This might have been caused due to antisymmetric/symmetric stretching vibration of $CH_2$ of aliphatic hydrocarbon compounds (Deniz et al. 2018). A peak shift was found from 1388 $cm^{-1}$ (pulp) to 1447 $cm^{-1}$ in 500 μm particles size powder and 1446 $cm^{-1}$ in 212 μm particles size powder, while both powders had almost similar peak. The shift of peaks at various peak intervals may be due to breaking of cellulose - hemicellulose intramolecular hydrogen bonds which form new amorphous cellulose and soluble saccharides (Zhao et al. 2015). The absorption bands at 1058, 1045 and 1039 $cm^{-1}$ of pulp, 500 μm and 212 μm particles size powder, respectively corresponds to methoxyl groups and primary and secondary alcoholic groups. The occurrence of oxy absorptions (such as phosphorus-oxy, nitrogen-oxy, sulfur-oxy, and silicon-oxy) between 1230 to 500 $cm^{-1}$ band were was highly dense in the overlapped region (Coates 2000).

**Table 3: FTIR analysis of aonla pulp and powder**

| Pulp | 500 (μm) | 212 (μm) | Assignment |
|---|---|---|---|
| 642,505.25 | 644, 522 | 638, 501 | C-I stretching, halo compound |
| 1058 | 1045 | 1039 | C-O stretching, primary alcohol |
| 1243.46 | 1247 | 1241 | C-O and C-N stretching; alkyl aryl ether and amine |
| 1388 | 1447 | 1446 | |
| 1633 | | 1629 | N-H bending, amine |
| 1741 | 1747 | 1749 | C=O stretching, esters |
| 2944 | 2929,2909 | 2857 | C-H stretching, alkane |
| 3446,3756 | 3457 | 3428 | O-H stretching, alcohol |

**Table 3: FTIR analysis of aonla pulp and powder**

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| 1741 | 1747 | 1749 | C=O stretching, esters |
| 2944 | 2929,2909 | 2857 | C-H stretching, alkane |
| 3446,3756 | 3457 | 3428 | O-H stretching, alcohol |

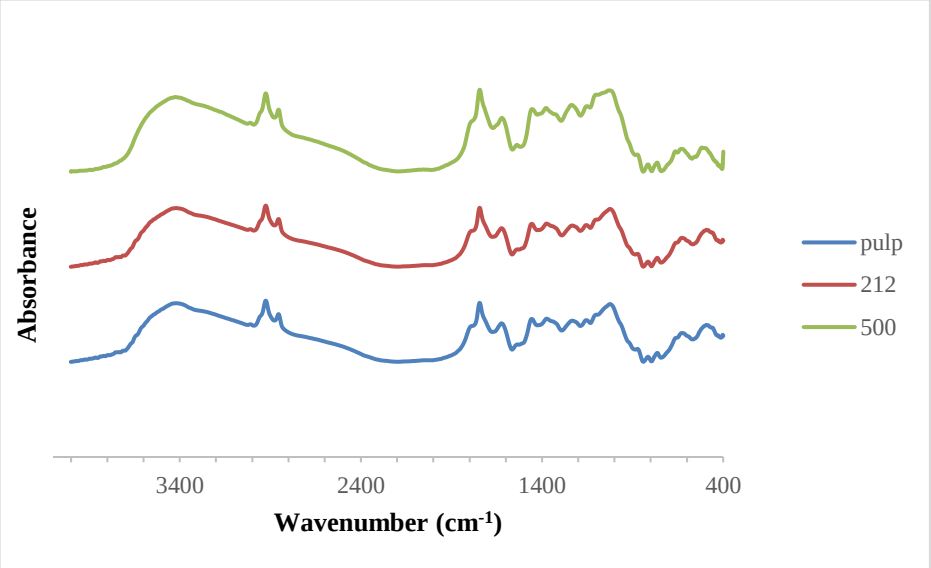
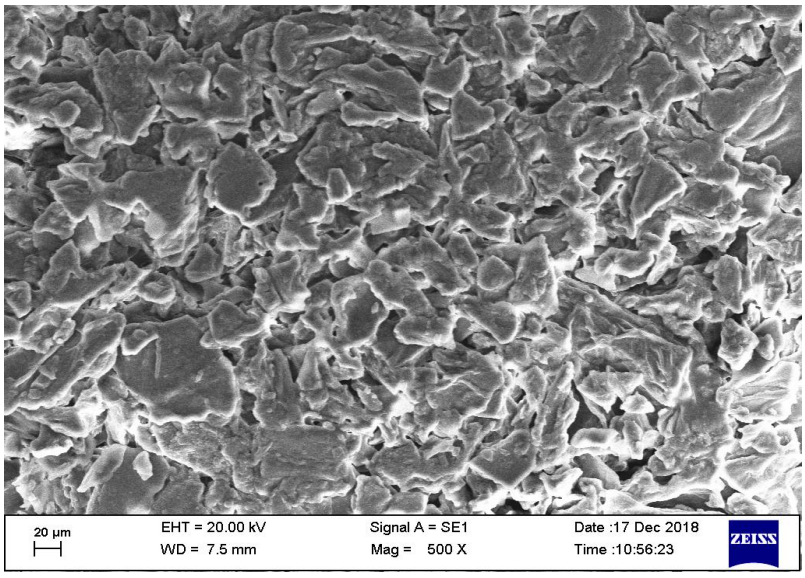


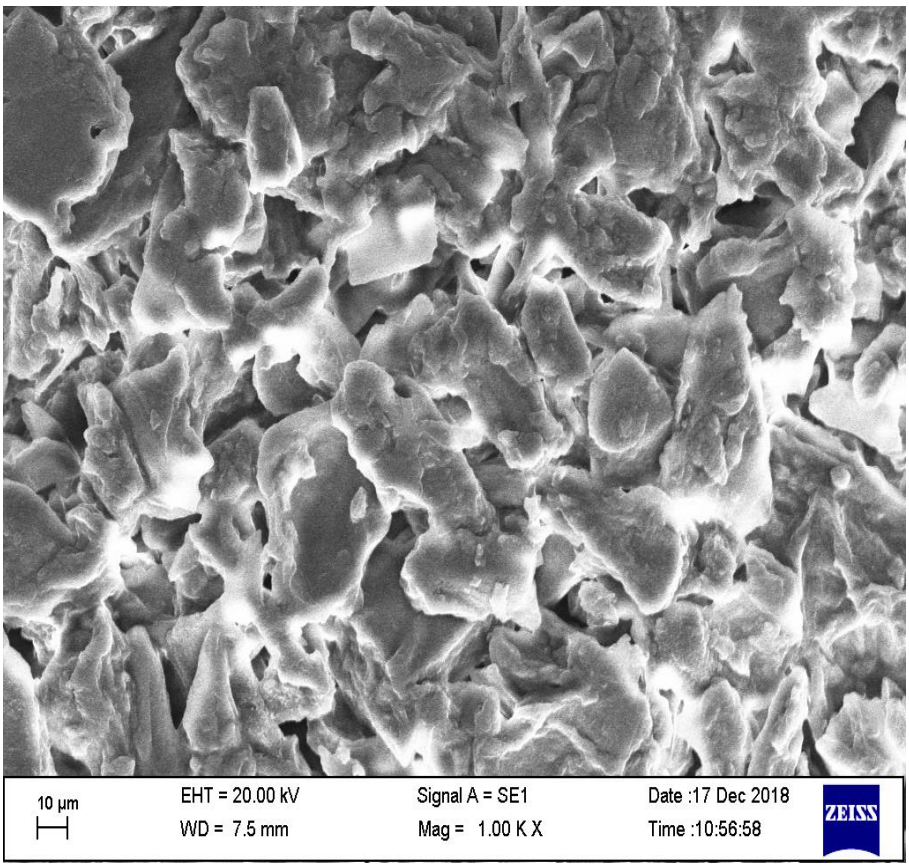

**Fig. 4:** Plot of FT-IR for Aonla pulp and powder (particles size 212 µm and 500 µm)

### 3.7 SEM Analysis

Aonla powder flakes after RW drying shows uniform thickness. The microstructure of aonla powder (212 µm) was studied using SEM analysis which shows that RW dried aonla powder particles had smooth upper surface and uniform thickness (Caparino et al. 2013; Shende and Datta 2019), as shown in Fig 5 (a) and Fig. 5(b). Shende and Datta 2019 had observed that mango pulp dried using RW drying produced powder particles having irregular shape, uniform thickness with smooth surface. However, oven drying, freeze and tray drying process produces mango powder particles of irregular, crinkled and corrugated surface with non-uniform thickness and uneven shape (Caparino et al. 2013; Afonso et al. 2019; Shende and Datta 2019). Similar results of unvarying thickness of powder particles were observed after RW drying of mango puree (Caparino et al. 2013; Shende and Datta 2019), haskap slurry (Celli et al. 2016) and probiotic powders of *Lactobacillus plantarum* (Yoha et al. 2019).




(a)




(b)

**Fig 5:** SEM analysis of Aonla powder at (a) 500 X and (b) 1 KX

## 4. Conclusions

RW drying of 3 mm thick aonla pulp layer was done to produce powder. It was observed that most of the drying occurs in the falling rate period and the whole process took 36 min for 81.52 % (wb) of pulp to produce 4.36 % (wb) of powder. The colour study shows that RW dried powder was lighter, less green and more yellow as compared to pulp before drying. Total colour difference for powder as compared to pulp shows that RW drying retains the colour of the drying material. Thermal properties study for pulp and RW dried powder shows that with decrease in moisture content thermal diffusivity, specific heat capacity and thermal conductivity decrease. The size distribution analysis of powder shows that more than 60% of the powder sample fall in the range of 355-212 µm size, 12.51 and 12.7 % in >150 µm and >355 µm, respectively. The oversize (>710 µm) particles retained were 2.47 % and undersize (<150 µm) particles were 2.30 % of powder sample. FT-IR analysis shows that no new chemical groups were found in RW dried aonla powder with comparison to pulp and shift of peaks being more pronounced for 212 µm powder particles than 500 µm powder particles with respect to pulp. This shift of peaks may be due to the formation of new amorphous cellulose and soluble saccharides after breaking of cellulose - hemicellulose intramolecular hydrogen bonds. SEM analysis of RW dried powder shows that the particles have uniform thickness and smooth upper surface.

## 5. Acknowledgements

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