Sustainability, Agri, Food and Environmental Research, (ISSN: 0719-3726), 11(X), 2023:
http://dx.doi.org/10.7770/safer-V11N1-art2913

Characterization and Property Analysis of Starch from Broken Parboiled Rice. Caracterización y Análisis de Propiedades del Almidón de Arroz Sancochado Quebrado

PA. Raajeswari[1*], S.M. Devatha[2], R. Pragatheeswari[2]

1- Associate Professor, Department of Food Science and Nutrition, , Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore, India

2- Ph.D Scholar, Department of Food Science and Nutrition, Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore, India

Corresponding Author: raajeswari_fsn@avinuty.ac.in

ABSTRACT

Rice (*Oryza sativa K.*) is the primary food consumed by half of the world's population and it provides 40-60 per cent of energy in daily diet. Production and consumption is anticipated to grow steadily every year. Rice is the rich source of starch, a polysaccharide consisting of amylose and amylopectin. Broken rice is the by-product and a key indicator of rice quality. Nearly 14 per cent of rice is broken while milling and during threshing process. The present study was carried out, to compare the properties and utilization of broken parboiled rice with whole rice. Alkaline extraction process is the best extraction method to isolate starch from broken rice that yields up to 70% of total starch with fewer residues. The characterisation of extracted starch from broken parboiled rice was investigated by Fourier-Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Thermo Gravimetric Analysis (TGA), Field Emission Scanning Electron Microscopy (FESEM). The quality of starch from broken rice estimated through physical, thermal and physiological properties show a similar property as like starch extracted from rice. It is the colourless and odourless compound which doesn't affect the colour and flavour of the product to be added that can be easily used in food industries at low cost. This study paves a way to utilize broken rice, a food industrial by-product and agro residue in an effective manner.

Key words: broken rice starch, property analysis, industrial by-product, agro residue, characterization, alkaline extraction.

RESUMEN

El arroz (*Oryza sativa* K.) es el principal alimento consumido por la mitad de la población mundial y proporciona del 40 al 60 por ciento de la energía en la dieta diaria. Se prevé que la producción y el consumo crezcan constantemente cada año. El arroz es una rica fuente de almidón, un polisacárido que consiste en amilosa y amilopectina. El arroz partido es el subproducto y un indicador clave de la calidad del arroz. Casi el 14 por ciento del arroz se rompe durante la molienda y durante el proceso de trilla. El presente estudio se realizó, para comparar las propiedades y aprovechamiento del arroz sancochado quebrado con el arroz integral. El proceso de extracción alcalina es el mejor método de extracción para aislar el almidón del arroz partido que produce hasta el 70 % del almidón total con menos residuos. La caracterización del almidón extraído del arroz sancochado partido se investigó mediante espectroscopia infrarroja transformada de Fourier (FTIR), difracción de rayos X (XRD), análisis termogravimétrico (TGA), microscopía electrónica de barrido de emisión de campo (FESEM). La calidad del almidón del arroz partido estimada a través de las propiedades físicas, térmicas y fisiológicas muestra una propiedad similar a la del almidón extraído del arroz. Es el compuesto incoloro e inodoro que no afecta el color y el sabor del producto que se va a agregar y que se puede usar fácilmente en las industrias alimentarias a bajo costo. Este estudio allana el camino para utilizar arroz partido, un subproducto industrial alimentario y residuo agrícola de manera efectiva.

Palabras clave: almidón de arroz partido, análisis de propiedades, subproducto industrial, agroresiduo, caracterización, extracción alcalina.

INTRODUCTION

Rice (*Oryza sativa* L.), queen of cereals contributes approximately 50 per cent of energy source of a human's daily diet. Approximately 480 million metric tons of milled rice is produced annually. China and India accounts about 50 per cent of the rice cultivation and consumption. It is rich in carbohydrate, moderate amount of protein and fat, and minimal source of vitamin-B complex such as thiamine, riboflavin and niacin. Rice starch is mainly composed of amylose and amylopectin and constitutes 12% water, 75%–80% starch and only 7% protein with 93% and 74% of digestibility and biological value. It is usually consumed after milling and refining process, in which most quantity of phytochemicals, lipids, dietary fibres and vitamins are removed, leaving, starch as the main compound. (Verma & Srivastav 2017).

Dead rice, brokens and brewers percentages, defectives foreign matter, presence of paddy, whiteness, chalkiness are the quality indicator of rice milling. Producers, traders and state and central warehousing corporation stores rice in large quantities to provide uniform supply throughout the year, to reserve and supply during natural calamities and to speculate on a good price either in domestic or in the export market which also paves way for more grain loss. (Tamil Nadu Agricultural University website, 2021)

Quality of the cereal grain is mostly deteriorated because of poor storage and transporting facility. The maximum storage capacities for cereal grains by farmers have been up to 1.5–2.0 tonnes. Around 30% of wheat and paddy produced every year is procured by the Food Corporation of India (FCI) and most of it is stored in warehouses. The agro-based industries like rice mills stores up to 1,000 – 20,000 tonnes of grains to meet their processing requirements (Dhingra D, 2016). Marketable Surplus and Postharvest Losses of Paddy in India, 2002, Directorate of Marketing and Inspection, Nagpur estimated Postharvest Losses of Paddy at the Producers' Level were 0.79, 0.89, 0.48, 0.16, 0.40 per cent during transportation from field to threshing floor, threshing, winnowing, transportation from threshing floor to storage, storage respectively with a total grain loss of 2.72 per cent and these losses end up in production of broken and poor quality rice during milling (Basavaraja H et al., 2007 & Kannan E, 2014).

110 million tons of paddy were produced in India in the year 2017–2018 and 7.5 per cent was broken during polishing. It is very cheap and is mainly used to extract ethanol and to feed cattle. Mondel. P *et al.,* (2021) stated that broken rice can also be utilised for starch extraction as it embodies 80 per cent of starch at 75–100 °C by alkaline extraction. Mazumder S & Bera D, 2013 revealed that production of malto-dextrin from broken rice is very beneficial because of its low cost and easy availability in India as well as in West Bengal. De Souza Schneider *et.al.,* 2018 analysed an average starch concentration of 80.1 per cent in the broken rice, that convert into glucose on hydrolysis, also stated that it cost effective by-product of paddy milling industry and a very good source of carbohydrates with low fat. Sydow Z.& Bieńczak K (2018) found that starch is a natural fiber that can be utilized in the packaging industry. It can also be used in the pharmaceutical and food industries as well.

MATERIAL AND METHODS

Materials: Broken parboiled rice variety was collected from nearby rice mundies around Coimbatore, Tamil Nadu, India which was the residue or by-product from milling and polishing industry. Figure 1a and 1b depicts the images of b

Figure 1. 1 a. Broken rice 1.b. Rice starch

Rice Starch Isolation: Starch was isolated by slightly modified alkaline extraction technique of Puchongkavarin *et al.,* (2005). Alkaline is used to remove the protein bounded to starch molecule as rice protein are soluble in alkaline medium and is neutralised by low acid medium. Schematic Diagram of rice starch extraction was represented in Figure- 2.

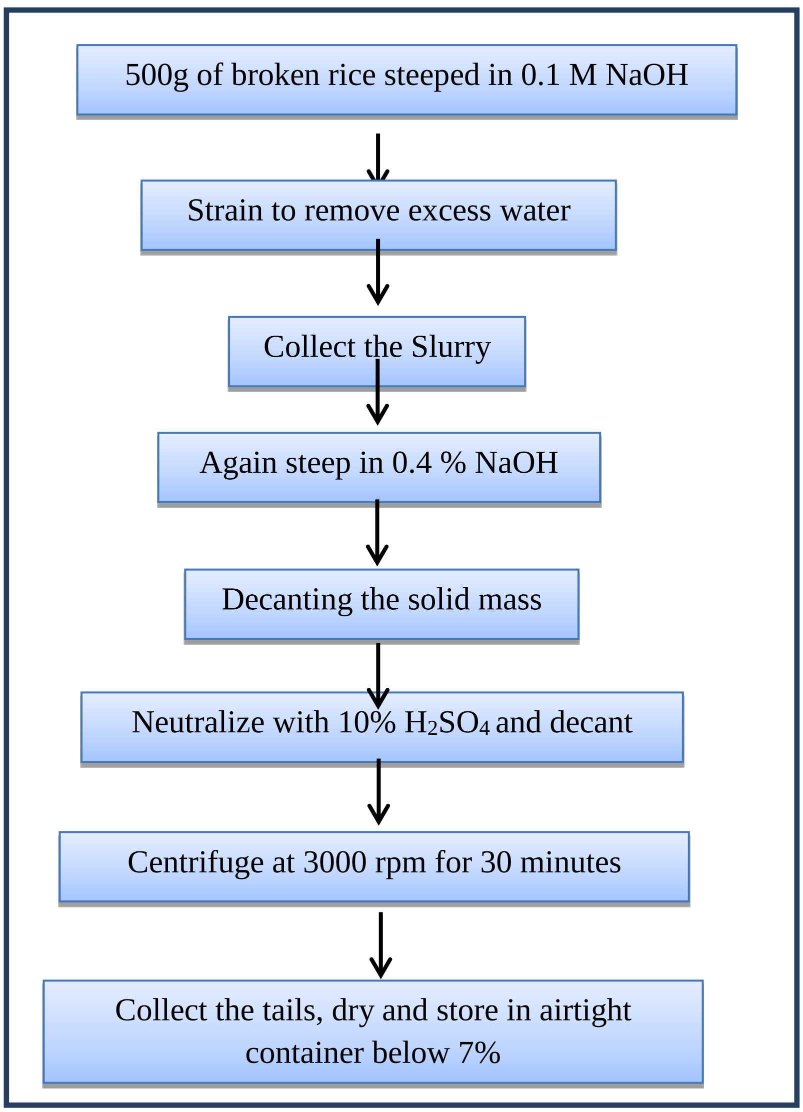

Figure- 2 Schematic Diagram of Rice Starch Extraction

3

500g of broken parboiled rice was steeped with gentle stirring in 0.1 M aqueous sodium hydroxide solution (0.4 % w/w NaOH) in the ratio of 1:2 for 18 h at room temperature followed by wet milling after filtering the 0.1 M NaOH solution. During milling a steady stream of tap water was added to prevent heating and gelatinisation of the starch. The slurry was then separated by an industrial scale strainer to filter excess amount of water added during wet milling. Slurry was collected and again steeped in 0.4% NaOH and kept undisturbed for 1 hour. Again, the slurry was decanted with free flow of water to decrease the alkalinity and to remove rice protein and fine fibre. Filter the solution to obtain solid mass and it is neutralized with 10% H_2SO_4 and separated by decanting. Decanting process repeats' till the solid mass attains neutral pH and centrifuged using Remi Laboratory centrifuge R8C at 3000 rpm for 30minutes. Repeat centrifugation till the rice starch tailed down. Supernatant was removed and starch sedimentation at the bottom was carefully collected and dried at room temperature.

Physical and Chemical Properties

Starch yield: Percentage of Starch yield is calculated by the initial weight of the broken rice to the dried starch extracted divided by 100.

% of Starch = Intial (wt) – Final (wt) × 100

Moisture Analysis: Moisture content of the extracted starch was analysed using Shimadzu Moisture Balance MOC – 120. As lower the moisture percentage, starch can be stored for longer duration. 2g of the sample was kept in the Moisture Balance at 120°C to estimate the percentage of moisture content in rice starch.

Ash Content: Ash content shows the quantity of micronutrients present in the food compound. It is estimated by using Muffle Furnace. Take the initial weight of crucible and lid. 3g of rice starch was measured and charred for 30 minutes and kept in muffle furnace at 600°C for 4 hours. If the sample is not greyish repeat the process till concordant value is obtained. Cool down it, in the desiccator at 30°C before weighing the sample. (AOAC, 2000).

Ash (%) = Weight of Ash / Weight of Sample × 100

pH value: pH value of the starch was estimated as rice starch was extracted by alkaline medium. It is measured by using digital pH meter. Calibrate the pH meter with 5 and 9 pH capsules. Stir the starch well with water and measure the acidity of the sample.

Field Emission Scanning Electron Microscope: Structure of Rice starch is measured by Field Emission Scanning Electron Microscope with energy dispersive X-ray spectroscopy (Tescan – Mira3 XMU, FESEM-EDX) at different ranges from 200µm to 10µm operated at 1.0 kV to determine the elemental composition of the samples. As the samples are electrically non-conducting, it is coated with gold nano-particles less than 10nm thickness which does not add any unwanted peaks to the X-ray spectrum and provide a path for the incident electrons to flow to ground.

X-Ray Diffraction: Crystallinity of starch is determined by X-ray diffraction (XRD) patterns using X – Pert Pro, PANalytical. It shows whether the sample taken is amorphous or crystalline in nature.

Fourier-transform infrared (FTIR) spectrophotometer: Functional property of broken parboiled rice starch was analysed by Shimadzu Fourier-transform infrared (FTIR) spectrophotometer in the range from $4000^{-1}$to 450 cm^{-1}.

Thermo Gravimetric Analyser (TGA): Thermal analysis of rice starch is identified by Thermo Gravimetric Analyser (EXSTAR/C300). When starch is heated, it undergoes certain physical and a chemical change with change in temperature is studied through TGA.

RESULT AND DISCUSSION

The results of the starch extracted from broken parboiled rice are discussed.

Physical and Chemical properties: Table.1 debits the physical and chemical properties of the starch extracted from broken rice.

Table.1 Physical and Chemical properties

S.No	Physical and Chemical properties	Value
1	Starch yield (%)	62
2	Moisture Analysis (%)	7
3	Ash Content (%)	0.2
4	pH value	6.4

Starch yield: 500g of broken parboiled rice yields 310 g of starch through alkaline extraction procedure that contributes a total percentage of 62. Puchongkavarin *et al.,* (2005) studied, rice starch extracted by 0.03–0.05 M NaOH solution yields 73–85% of starch, 0.07–0.42% of residual protein and 0.07–2.6% of damaged starch.

Moisture Analysis: Moisture content of the starch was determined by digital moisture balance and it contains 7 per cent and stored in air tight container. Janaun *et al.,* (2016) found out as the moisture content in rice starch decreases, amylose content increases and <8-12 % is the optimum moisture content to store the extracted starch for a longer duration.

Ash content: Ash content of the alkaline extracted broken rice starch is 0.2% which shows minimal residue. Koh, S. P., & Long, K. (2012) studied that the ash content of broken rice starch was 0.07±0.01% through centrifugation at laboratory scale.

pH Value: pH value of the broken rice starch is 6.4 which is merely alkaline to neutral. Usman *et al.,* (2014) experimented rice starch extraction with different pH parameters and concluded alkaline extraction of 9.5 pH is the best method for broken rice with a maximum yield of 85 to 95% for all temperature.

Field Emission Scanning Electron Microscope: Rice starch is measured in Field Emission Scanning Electron Microscope with energy dispersive X-ray spectroscopy at different ranges from 200µm to 10µm along with elemental mapping as given in Figure 3. a, 3.b, 3.c. The granular size of starch extracted from broken rice is less than 10 µm in diameter with a smooth surfaced regular polyhedral structure. The chemical formula for starch is $(CH_2O)_{n.}$ The composition of starch particles is determined by Energy dispersive X-ray Spectroscopy showed 49% Carbon and 51% Oxygen. Cao *et al.,* (2020) identified native rice starches granules displayed regular polyhedrons with distinct edges and smooth surfaces, and there were no pinholes noticed on the particles. Corgneau *et al,* (2019) observed rice starch granules were very small (<10 µm diameter) with some agglomerates and displayed an intermediate amylopectin content (between 78% and 80%).

X-Ray Diffraction: Crystalline structure, crystal plane and crystal size of the broken rice starch is determined by X-Ray Diffraction. Figure 4 represents the X-Ray Diffraction peaks of starch with the concentration on the diffraction angle 2θ of 15°-24°, and strong diffraction peaks appeared at the diffraction angles 2θ at 15.34°, 17.17°,18.29° and 23.16° corresponding to the (1 0 0), (1 0 1), (2 1 4) and (1 1 0) planes of starch with the intensive height of 257, 294, 323, 300 and relative intensity of 79.62, 91.02, 100 and 92.96 respectively and displayed a typical A type diffraction pattern (Table 2).

The average crystallite size of broken rice starch has been estimated using the Debye-Scherrer formula as

$$D = K \lambda / \beta \cos\theta$$

where, D is crystalline size, nm, λ is X-ray wave length of Cu, β is full width at its half maximum and k is Dimension less shape factor, 0.94. From the Debye-Scherrer formula, the Crystal size of the starch extracted is between 10.55 nm to 15.71 nm with an average grain size of 12.83nm. This pattern matches with the study of Cao *et al.,* (2020) and Matmin *et al.,* (2018), with concentrated on the diffraction angles 2θ between 10°-27°, and strong diffraction peaks appeared at 15°, 17°, 18°, and 23° that forms type A diffraction pattern. The relative crystallinity of rice starch is 26.75%. The A-type crystalline structure has a large hollow structure, which was formed by forming an inclusion complex between linear short chain and polar organic molecules.

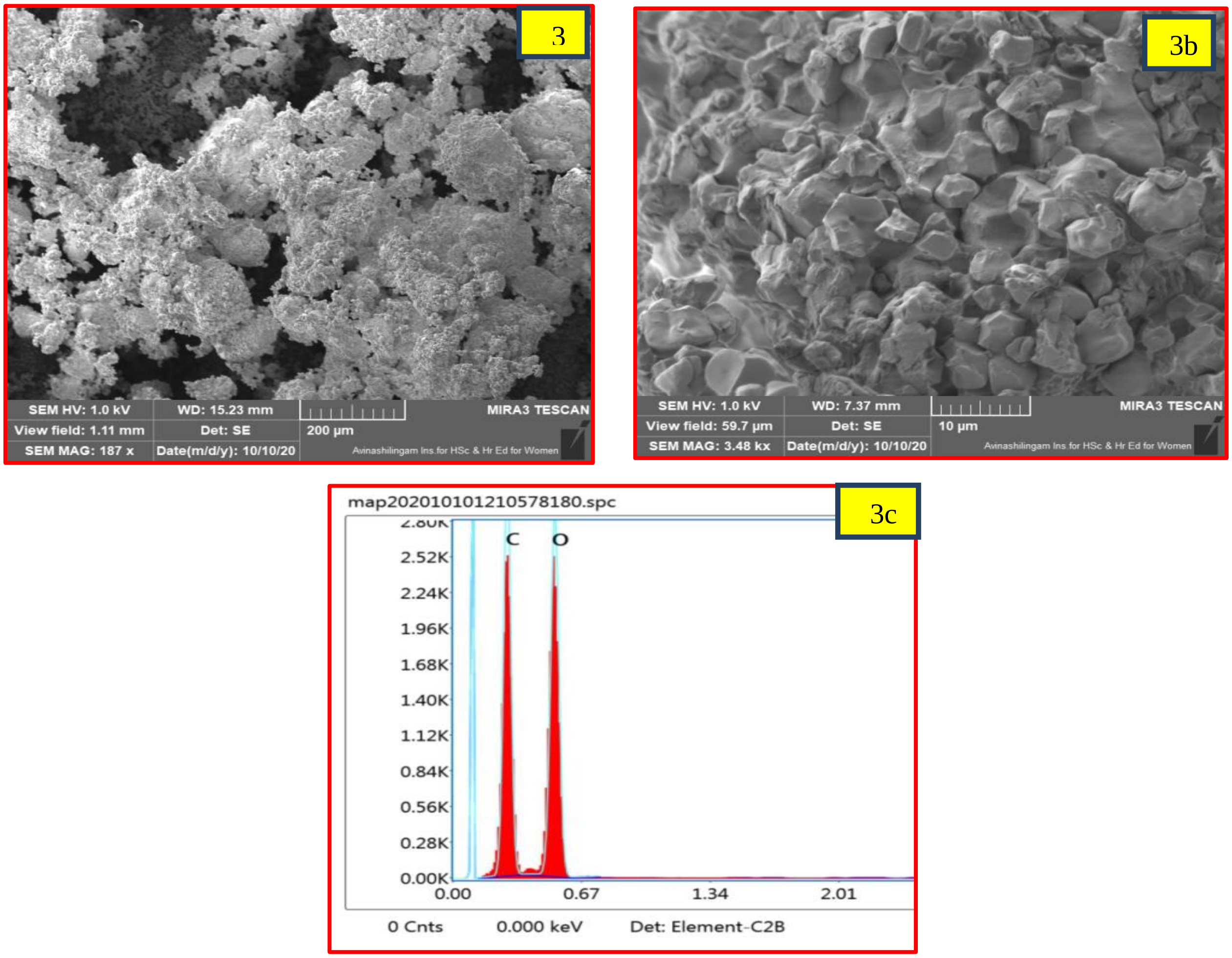

Figure 3. Broken Rice Starch images from Field Emission Scanning Electron Microscope with energy dispersive X-ray spectroscopy 3.a.200 µm, 3.b. 10 µm, 3.c. composition

Table -2 .X- ray Diffraction Results

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
15.3495	257.59	0.6691	5.77268	79.62
17.1757	294.49	0.6691	5.16278	91.02
18.2926	323.54	0.5353	4.85001	100.00
23.1661	300.78	0.8029	3.83955	92.96

Sustainability, Agri, Food and Environmental Research, (ISSN: 0719-3726), 11(X), 2023:
http://dx.doi.org/10.7770/safer-V11N1-art2913

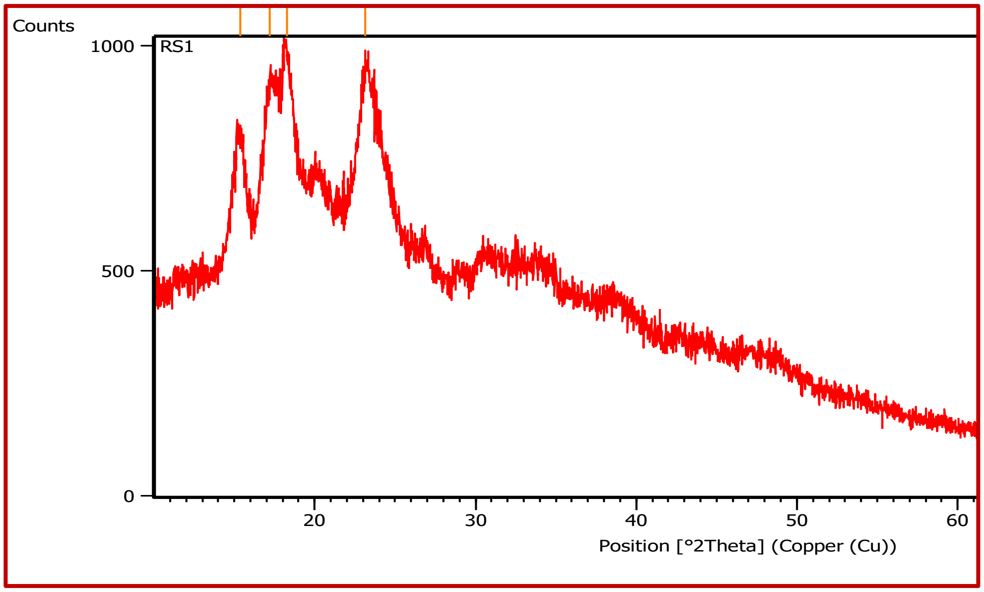

Figure 4 X-Ray Diffraction pattern of Broken Rice Starch

Fourier-Transform Infrared (FTIR) spectrophotometer: Functional properties of rice starch were analysed by using Fourier-transform infrared (FTIR) spectrophotometer in the range from 4000 cm^{-1} to 450 $cm^{-1}$was presented in Figure 5. Peaks at 1527 and 2360 cm^{-1} were attributed due to C-H stretches, a strong peak was obtained at 1010 cm^{-1} was assigned to the anhydro glucose ring of the O–C stretch. Weak peaks of vibrational bands in the fingerprint region, at 408, 455, 547, 601, 702, 756 and 856 cm^{-1} due to the vibrations of pyranose ring in the glucose unit and confirmed the presence of glucose polymer and confirmed the presence of carbohydrate. Some other weak peaks like 925, 1010, 1080 and 1149 cm^{-1} were attributed to C–O stretching. Hence, from the functional properties of rice starch by FTIR shows the presence of abundant quantity of glucose molecule. Matmin *et al.,* (2018) also investigated on rice starch and attained different peaks between 3000 to 400 cm^{-1} and confirmed the presence of intact glucose polymer by the skeletal mode vibrations at different region (Table 3).

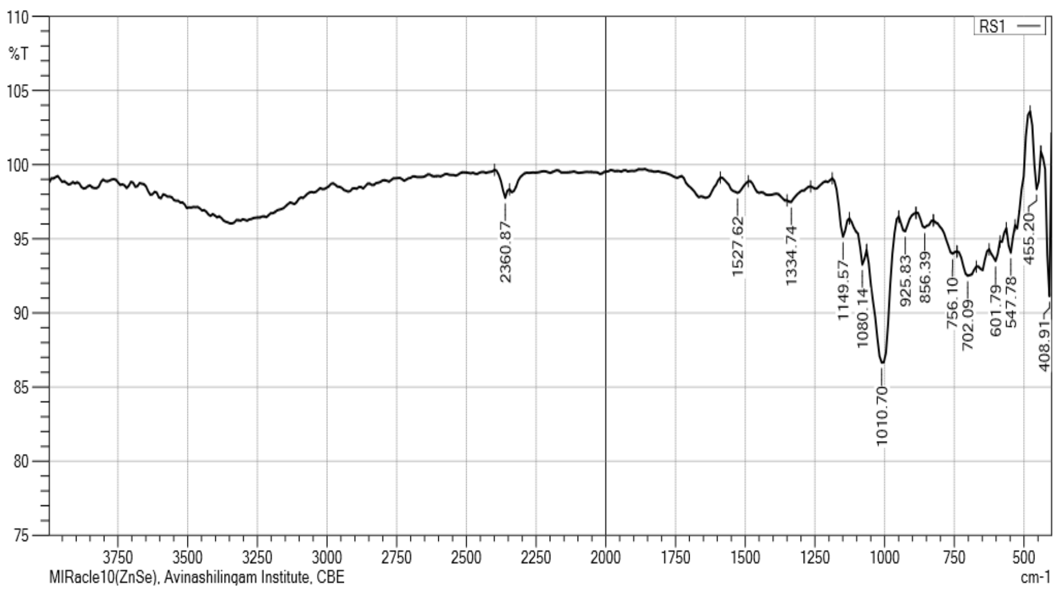

Figure 5 Fourier-Transform Infrared (FTIR) spectrophotometer Peak of Broken Rice Starch

7

Table-3. Fourier-Transform Infrared (FTIR) spectrophotometer Data

Wavelength (cm^{-1})	Intensity of Peak	Absorbing feature
2360.87	Medium	O=C=O stretch
1650.17, 1527.62	Medium	C=C bend
1334.74	Weak	O-H bend
1149.57	Strong	C-C stretch
1080.14	Medium	C-O stretch
1010.70	Strong	C-O stretch
925.83, 856.39	Medium	C-C bend
756.10, 702.09, 601.79	Medium	CHO bend
547.70, 455.20, 408.91	Strong	CHO stretch

Thermo Gravimetric Analyser: Thermal property of the starch extracted from broken rice is estimated and given in Figure 6. which measures the change in weight during increase or decrease in temperature. DTA graph shows that, starch in dry form while heating it undergoes exothermic reaction as produces heat and started to char above 100°C. TGA shows, a total loss of 91.2 % that decompose slowly from 100°C and the total decomposition end at 1000°C. Initially, the temperature was slowly increased from 31°C to 300°C, the weight of the starch taken was decreased from 12.84mg to 9.35mg. From 300°C to 670°C, the decomposition rate was high and decreased from 9.35mg to 5.53 mg between, 670°C to 1000°C, the decomposition rate was decreased from 5.53 mg to 2.74 mg. The temperature slowly increased from 31°C to 1000°C and the total decomposition rate was 91.2% with the average starch weight loss of 12.84 mg to 1.13 mg as residual mass. As the concentration of starch increases, the moisture content decreases lead to weight loss. Rice starch is crystalline in dry basis and during gelatinization turns into amorphous due to O-H bond while heating. Starch readily undergoes retrogradation and syneresis while change in temperature.

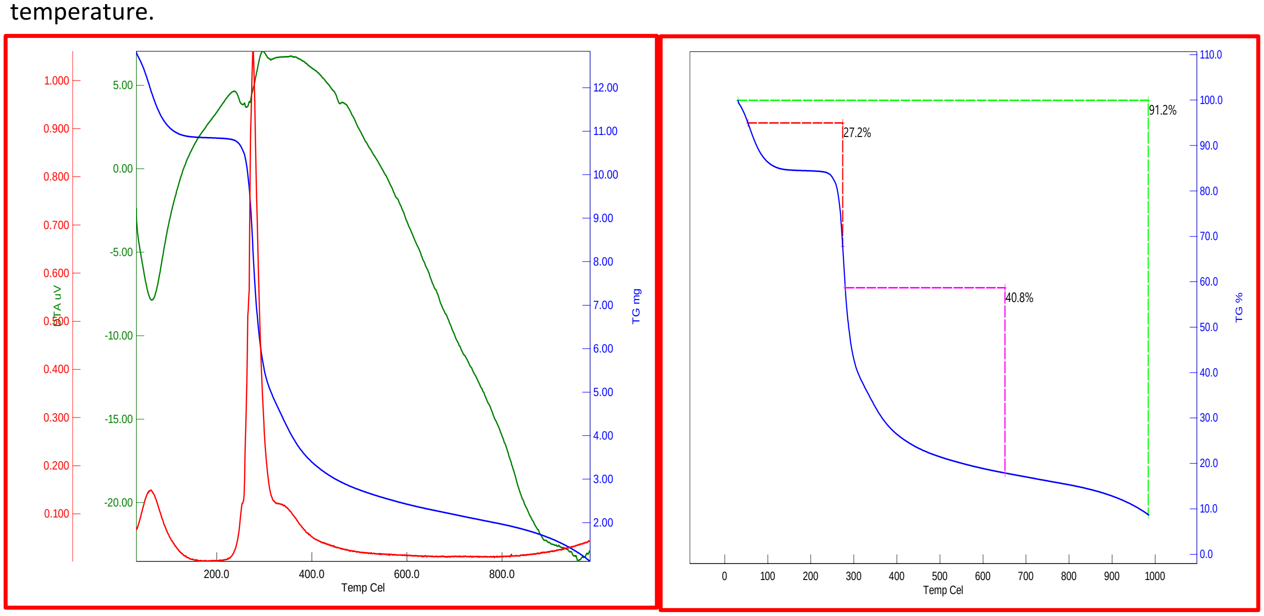

Figure 6 Thermo Gravimetric Analysis of Broken Rice Starch

As conclusion, paddy is one of the major crops cultivated, harvested and processed in India, which is consumed daily by half of the world's population. Broken rice is the by-product as well as agricultural residue during milling and manual or mechanical threshing with a total loss of 14-18 % of paddy production. Rice starch is composed of tiny granules (<10µm), white coloured and odourless that makes ideally suitable for many different food-based industries. The process of isolation and extraction of starch from broken rice is an easier method and paves a way for better utilization of underutilised raw material. This study provides an insight on the characteristics and properties of starch from broken parboiled rice that are similar to rice starch. Since, most of the broken rice is utilized in ethanolic extraction; this research suggests broken rice starch can also be used as a replacer for rice starch in industrial and hospitalization sector that has, all the similar properties.

ACKNOWLEDGEMENTS

The authors sincerely thank DST-SERB for giving us an opportunity to do this research project entitled “Edible Bio-Based Food Packaging with ‘Smart’ Approach towards ‘Econutrition’-A Commitment to Beat Single-Use Plastics”to make the universe free of plastics and Avinashilingam Institute of Home Science and Higher Education for Women, Coimbatore.

REFERENCES

AOAC, Official Methods of Analysis: Moisture and Ash, 15 ed., Association of Official Analytical Chemistry, Arlington, U.S.A., 777, 2000.

Basavaraja, H., Mahajanashetti, S. B., & Udagatti, N. C. (2007). Economic analysis of post-harvest losses in food grains in India: a case study of Karnataka. *Agricultural Economics Research Review*, *20*(347-2016-16622), 117-126.

Cao, C., Shen, M., Hu, J., Qi, J., Xie, P., & Zhou, Y. (2020). Comparative study on the structure-properties relationships of native and debranched rice starch. *CyTA-Journal of Food*, *18*(1), 84-93.

Corgneau, M., Gaiani, C., Petit, J., Nikolova, Y., Banon, S., Ritié-Pertusa, L & Scher, J. (2019). Digestibility of common native starches with reference to starch granule size, shape and surface features towards guidelines for starch-containing food products. *International Journal of Food Science & Technology*, *54*(6), 2132-2140.

De Souza Schneider, R. D. C., Junior, C. S., Fornasier, F., de Souza, D., & Corbellini, V. A. (2018). Bioethanol production from broken rice grains. *Interciencia*, *43*(12), 846-851.

Dhingra, D. (2016). Evolution and trends in food grain storage in India. In *Proceedings. 10th International conference on controlled atmosphere and fumigation in stored products (CAF 2016), CAF Permanent Committee Secretariat, Winninpeg, Canada* (pp. 47-52).

Koh, S. P., & Long, K. (2012). Comparison of physical, chemical and functional properties of broken rice and breadfruit starches against cassava starch. *Journal of Tropical Agriculture and Food Science*, *40*(2), 211-219.

Janaun, J., Kong, V. V., Toyu, C. G., Kamin, N. H., Wolyna, P., & Lee, J. S. (2016, June). Effect of moisture content and drying method on the amylose content of rice. In *IOP Conference Series: Earth and Environmental Science* (Vol. 36, No. 1, p. 012064). IOP Publishing.

Matmin, J., Affendi, I., Ibrahim, S. I., & Endud, S. (2018). Additive-free rice starch-assisted synthesis of spherical nanostructured hematite for degradation of dye contaminant. *Nanomaterials*, *8*(9), 702.

Mazumder, S., & Bera, D. (2013). Production of malto-dextrin from broken rice. *International Journal of Research in Engineering and Technology*, *2*(10).

Mondal, P., Sadhukhan, A. K., Ganguly, A., & Gupta, P. (2021). Optimization of process parameters for bio-enzymatic and enzymatic saccharification of waste broken rice for ethanol production using response surface methodology and artificial neural network–genetic algorithm. *3 Biotech*, *11*(1), 1-18.

9

Puchongkavarin, H., Varavinit, S., & Bergthaller, W. (2005). Comparative study of pilot scale rice starch production by an alkaline and an enzymatic process. *Starch-Stärke*, *57*(3-4), 134-144.

Sydow, Z., & Bieńczak, K. (2018). The overview on the use of natural fibers reinforced composites for food packaging. *Journal of Natural Fibers*.

Usman, M., Ishfaq, M. T., Malik, S. R., Ishfaq, B., & Iqbal, M. (2014). Effects of Temperature, pH and Steeping Time on the Extraction of Starch from Pakistani Rice. *Int J Scientific Engr Res*, 877-892.

Verma, D. K., & Srivastav, P. P. (2017). Proximate composition, mineral content and fatty acids analyses of aromatic and non-aromatic Indian rice. *Rice Science*, *24*(1), 21-31.

Kannan, E. (2014). *Assessment of pre and post harvest losses of important crops in India*. Bengaluru, India: Agricultural Development and Rural Transformation Centre, Institute for Social and Economic Change.

http://www.agritech.tnau.ac.in/expert_system/paddy/phtc.html (accessed on 01.08.2021]

Received: 22th Jule 2022; Accepted: 06th September 2022; First distribution: 05th October 2022.