



Research Article

A comparative study on physical characteristics of cellulose isolated from various sources of rice husk

Talasila Gopalakrishna Murthy* , Gummadi Priyanka, Sayala Hema Mani kumara, Murari Sri Mani Deep, Gurram Charanya and Ramagiri Yagna

Department of Pharmaceutics, Bapatla College of Pharmacy, Bapatla-522101, Andhra Pradesh, India.

Abstract

Cellulose is the biomass that is most easily found in plants, bacteria, and marine organisms. However, it is not found in pure conditions in nature and is always bound to other materials such as lignin, hemicellulose, silica, wax, and ash. Rice husk is an abundant agricultural waste and has high cellulose content from different rice husks such as Pusa Rh-10 (92), Dwarf (22-70), Parvati (27-16), Sona and BPT that have been successfully isolated. The cellulose obtained from different sources was characterised by various physical, micromeritic properties and percent yield. Kawakita plots were used to observe micromeritic properties. The percentage yield and other properties were found to be dependent on the source of cellulose. The % yield was found to be from 33% to 60%. The flow properties such as carr's index, bulk density and hausner ratio were found to be dependent on the source of cellulose and the particle size of cellulose. Among the varieties used in this investigation, 92 varieties showed good flow property and produced a high yield of cellulose.

Article Information

Received: 28 November 2024

Revised: 26 December 2024

Accepted: 30 December 2024

Published: 31 December 2024

Academic Editor

Prof. Dr. Christian Celia

Corresponding Author

Prof. Dr. Talasila

Gopalakrishna Murthy

E-mail: gopalakrishnatalasila@

yahoo.com

Tell: +919912342094

Keywords

Cellulose, rice husk, physical characters, Kawakita plots.

1. Introduction

The utilization of waste materials plays a vital part in solving economic issues at present. Rice husk is the major agro-waste from rice production [1]. Due to its tough structure and huge bulk, rice husk has limited applications and was treated by burying underground or open field burning. The International Grains Council (IGC) has released data that projects global rice production to reach 511.3 million tons in 2023/24, resulting in the production of 102.3 million tons of rice husk waste. The lignocellulosic part of the rice husk can be utilized to produce value-added products such as fuel, chemicals, etc., while silica can be utilized to produce silicon-based materials. It is of great benefit to both the economy and the environment to effectively convert rice husk into

valuable products such as biosilica and biofuels [2]. Rice husk, the protective outer layer of the rice kernel, has been regarded as a waste product for a very long time. However, they can be a great fuel for bioenergy production due to their high cellulose and lignin content. Rice waste management holds immense potential for a sustainable future. The rice industry can be more resource-efficient and holistic if byproducts are perceived as valuable resources. So this study is aimed at imparting value addition to rice husk by converting cellulose as a pharmaceutical additive. Studies have shown that rice husk contains about 35–40% cellulose, 15–20% hemicellulose, and 20–25% lignin [3]. Johar et al. [4] successfully produced cellulose fibers and nanocrystals from rice

husk using an acid hydrolysis treatment. Non-ionic cellulose ethers such as ethyl cellulose (EC), hydroxyethyl cellulose, hydroxypropyl cellulose (HPC), methyl cellulose (MC), carboxymethyl cellulose (CMC) or hydroxypropyl-methyl cellulose (HPMC) and anionic ether derivatives like sodium carboxymethyl cellulose (NaCMC) are useful as bioadhesives [5]. Cellulose ethers, alone or their mixtures with other polymers, have been studied in oral [6], buccal [7], ocular [8], vaginal [9] and transdermal [10] bioadhesives. Ether and ester derivatives of cellulose are widely used as coatings of solid pharmaceuticals. Specific release characteristics in pharmaceuticals to prepare various modified release drug delivery systems such as sustained release, delayed release, extended release, immediate release, pulsatile release, or step-by-step release dosage forms [11]. Rice husk consists of three sections: epidermis, sub-hypodermis and hypodermis. The thickness of these layers, the diameters of the hollow fibers and the wall thickness vary with the variety of rice husk. The elastic modulus is typically between 0.3 and 2.6 GPa, and the ultimate tensile strength varies from 19 to 135 MPa depending on the variety of rice husk. The physicochemical properties and the percentage yield of an isolated substance depends on biological and geographical source and hence there is a need to study the influence of such factors. So studies were conducted on the influence of the source of rice husk on cellulose properties.

2. Materials and methods

2.1. Chemicals

Glacial acetic acid (SDFCL, Mumbai, India), sodium hydroxide (Lobachemie, Mumbai, India), nitric acid (Qualigens, Mumbai, India), ethanol and methanol (Fisher Scientific, Mumbai, India).

2.2. Materials

Rice husk was collected from rice mills in Bapatla, Andhra Pradesh, India. Glassware: (Borosilicate glass).

2.3. Equipments

Disintegrator apparatus, weighing balance D8-8520, (Essac-Teraoka Limited, Bengaluru, India) Hot air oven (Thermolab, Mumbai), bulk density apparatus BD-7CC, (Campbell Electronics, Mumbai, India) and sieves (Shreeji, Mumbai).

2.4. Isolation of cellulose from different sources of rice husk

2.4.1. Cellulose extraction

After weighing precisely 5g of rice husk, it was transferred to a 250 mL Erlenmeyer flask containing 100 mL of 80% glacial acetic acid and 10 mL of 70% nitric acid. After sealing the flask in aluminium foil, it was heated for 20 minutes at 120 degrees Celsius. After allowing the sample mixture to cool, 60 millilitres of distilled water was added. The mixture was then filtered and cleaned using 95% ethanol and distilled water. The residue was dried for 19 hours at 60 degrees Celsius in an oven. The amount of cellulose extracted from various husks was computed [12,13].

2.4.2. Characterisation of cellulose

Determination of particle size: The particle size was determined by subjecting it to sieve analysis. Selected standard sieves were arranged in such a manner that the coarsest at the top and the finest at the bottom [14]. The sample was placed on the coarse sieve. The lid was tightly fixed and set on a mechanical stirrer for about 20 min. The mechanical shaker was switched on with its timer set at 20 min. The samples retained on each sieve were collected separately, weighed and the data were analysed to observe the size distribution by using the mathematical formula given in equation 1.

$$\text{Average diameter} = \frac{\text{sum of weight size}}{\text{sum of weight retained}} \quad (1)$$

2.5. Measurement of density

2.5.1 Bulk density

The bulk density of a powder is the ratio of the mass of a powder sample to its volume, including the interparticulate void volume and intraparticle void volume [15]. Hence, the bulk density depends on both the density of powder particles and, in particular on the voids in the spatial arrangement of particles in the powder bed. Bulk density is commonly expressed in grams per milliliter. The bulk properties of a powder are dependent upon the preparation, treatment and storage of the sample, i.e., how it has been handled. The particles can be packed to have a range of bulk densities. Therefore, the untapped bulk density and tapped bulk density are differentiated.

$$\text{Bulk density} = \frac{\text{Weight of powder}}{\text{Volume of powder}} \quad (2)$$

2.5.2. Tapped bulk density

The tapped bulk density is an increased bulk density

attained after mechanically tapping a receptacle containing the powder sample. The tapped bulk density is obtained by mechanically tapping a graduated measuring cylinder or vessel containing the powder sample. After observing the initial untapped bulk volume (V_0) and mass (m_0) of the powder sample, the measuring cylinder or vessel is mechanically tapped, and volume or mass readings are taken until little further volume or mass change is observed. The mechanical tapping is achieved by raising the cylinder or vessel and allowing it to drop, under its own mass, a specified distance [16].

Procedure: Carefully level the powder without compacting, and read the untapped bulk volume (V_0) to the nearest graduated unit. Secure the cylinder in the support. The powder sample of known particle size was subjected to 25, 50, 75, 100, 125 and 150 taps and the corresponding volumes V_{25} , V_{50} , V_{75} , V_{100} , V_{125} and V_{150} were recorded. If the difference between V_{125} and V_{150} is less than or equal to 2 mL, then V_{150} is considered as the tapped bulk volume. If the difference between V_{125} and V_{150} exceeds 2 mL, repeated in increments of, for example, 125 taps, until the difference between successive measurements is less than or equal to 2 mL. Calculated the tapped bulk density in grams per milliliter using the formula m/V_f (where V_f is the final tapped bulk volume).

2.5.3. True density

The true density refers to the density of the solid phase of the particles. It excludes the volume contribution of both inter- and intraparticulate spaces. Therefore, the true density of a powder is independent of powder porosity, compaction, and pretreatment of the sample [17].

Procedure:

True density of the cellulose samples isolated from different rice husks was determined by the solvent displacement method [18]. The true density of cellulose was determined by the liquid displacement method and the procedure was mentioned here. Weigh accurately a clean and dry density bottle, observe the weight of the density bottle with a small quantity of powder sample and water separately. Calculated the true density of the given powder sample by using the following equation. The experiment was repeated for various particle size

fractions.

$$\rho = \frac{m}{v} \quad (3)$$

Where,

ρ = Density

m = Mass

v = Volume

2.5.4. Carr's Index (C_i):

Tapped and bulk density measurements can be used to estimate the Carr's index of a material. Carr's index was determined by the following formulae [19].

$$\text{Carr' index} = ((\text{Tapped density} - \text{bulk density}) / \text{Tapped density}) \% \quad (4)$$

2.5.5. Hausner's Ratio:

Hausners ratio is a guide to ease of powder flow; it is calculated by the following formula.

$$\text{Hausner ratio} = \text{Tapped density} / \text{Bulk density} \quad (5)$$

2.5.6. Angle of repose

Angle of repose (α) was determined by fixed the funnel method. The blend was poured 5 g. through a funnel that can be raised vertically until a maximum cone height (h) was obtained. The radius of the heap (r) was measured and the angle of repose was calculated. It is used to determine the flow property of powder [20, 21].

$$\alpha = \tan^{-1} (h/r) \quad (6)$$

Percentage porosity: Porosity is the percentage of void space in a powder. It was defined as the ratio of the volume of the voids or pore space divided by the total volume. It is written as either a decimal.

$$n = \frac{V_{\text{pore space}}}{V_{\text{total}}} \quad (7)$$

2.5.7. Kawakita plots

Kawakita developed plots to study power densification using the degree of reduction in volume.

$$C = \frac{(V_0 - V_p)}{V_0} = \frac{abP}{(1 + bP)} \quad (8)$$

It can be arranged to give:

$$\frac{P}{C} = \frac{P}{a} + \frac{1}{ab} \quad (9)$$

Where,

v_0 is the initial volume of the powder bed and v_p is

Table 1. Physical characteristics of cellulose isolated from different sources of rice husk

Source (Rice variety)	Practical yield (%)	Particle size (cm)	Bulk density	Tapped density	True density	Carr's index	Hausner ratio	Porosity (%)
92	60	542.38	0.375	0.52	1.10	29	1.38	28.75
22-70	50	598.30	0.35	0.47	1.54	25	1.34	25
27-16	33	564.48	0.35	0.48	1.42	26	1.37	27
SONA	56	381.75	0.39	0.51	1.22	25	1.32	24
BPT	52.3	761.66	0.33	0.48	1.42	32	1.45	32

the powder volume after application of pressure P . a and b are constants which are obtained from the slope and intercept of p/c vs p plots.

The constant a is equal to the minimum porosity of the powder system prior to compression. In contrast, b is also known as the coefficient of compression, which is related to the plasticity of the material.

Values of $(1-a)$ give the initial relative density of the material p , which provides a measure of the initial packed density of tablets with the application of small pressure. The reciprocal of b gives a pressure term, p_k , which b is the pressure required to reduce the powder bed by 50%. For plastic materials, the value of p_k is inversely proportional to the degree of plastic deformation occurring during the densification process. The lower the value of p_k , the higher the degree of plastic deformation occurring during compression [22].

3. Results and discussion

Cellulose was isolated from different rice husks such as 92, 22-70, 27-16, Sona, and samba masuri (BPT). The cellulose was successfully collected from all these sources and the % yield of cellulose obtained from these sources was depicted in Table 1. The % yield was found to be from 33% to 60%. The yield of cellulose was lower in 27-16 variety and the % yield was found to be high in 92 varieties. Based on the percentage of yield of cellulose, these rice husks can be ranked as 27-16<22-70<BPT<Sona<92. So from the above studies it was observed that the percentage yield of cellulose is dependent on the source of rice husks. The particle size of cellulose was determined by using sieve analysis technique. The particle size observed from these materials was presented in Fig. 1. The particle size distribution followed a normal distribution and varied from one source of rice husk

to another and depended upon the source of the raw material. The micromeritic properties of the cellulose isolated from different sources are presented in Table 1. The bulk density was found to be from 0.33 - 0.3. The tapped bulk density was found to be from 0.47- 0.52 g/mL.

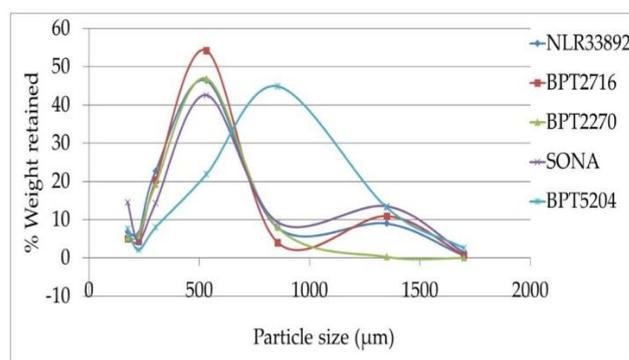


Figure 1. Normal distribution graph related to cellulose collected from different sources.

The micromeritic (flow) properties were expressed by Carr's index and observed Carr's index values were found to be 25-32. From these results, (Carr's index and Hausner's values are represented in Table 1). The flow property was found to be poor for 92, 22-70-passable, 27-16-poor, Sona-passable and BPT- very poor. Based on Hausner ratio these materials can be ranked as sona<22-70<27-16<92<BPT. The flow properties were also expressed as kawakita plots in Fig. 2 and the observed a and b values were presented in Table 2 and Fig. 2. From the observed results, they have poor flow ability and high cohesiveness. Thus these studies revealed that cellulose can be used to form good compacts. The flow properties may be improved by using the larger particle size fractions of cellulose. Such coarse cellulose can be utilized as a direct compressible diluent to formulate solid dosage forms such as tablets and capsules.

Table 2. Kawakita parameters observed from cellulose isolated from different sources of rice husk

Sl. No	Variety	Kawakita parameters	
		A	B
1.	92	3.56	0.280
2.	22-70	3.88	0.009
3.	27-16	0.33	0.040
4.	SONA	4.12	0.009
5.	BPT	3.16	0.012

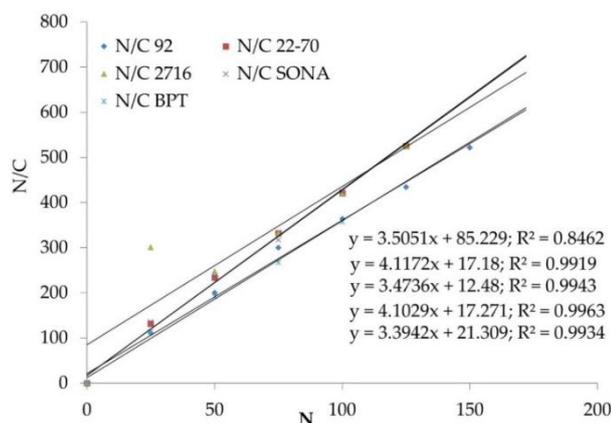


Figure 2. Kawakita plots related to cellulose collected from different sources.

The percentage porosity of the cellulose obtained from different sources of rice husk is calculated from the observing bulk density and true density. Observed % porosity values are shown in Table 1 and the highest % porosity was observed in BPT variety compared with the other materials. The high porosity may facilitate good disintegration and rapid dissolution and thus cellulose is suitable to prepare tablets with good compatibility and dissolution. The increase in the particle size resulted in improved flow properties of cellulose and was found to be suitable for direct compression.

4. Conclusions

The cellulose content was different from various rice husks available in the local market and popularised as 92, 22-70, 27-16, Sona and BPT. The particle size distribution and flow properties of cellulose are dependent on the source of rice husk. Careful selection of the rice husk is required to fulfil the desirable attributes. The passable flow properties obtained for 22-70 and sona are suitable for preparing

direct compressible tablets. The highest porosity observed in BPT facilitates the high dissolution and disintegration which is very helpful in the formulation of tablets containing low water soluble drugs. The result of this investigation suggests that the percentage yield and micromeritic properties of cellulose depend on the source of the rice husk. Screening of cellulose is essential to formulate direct compressible tablets and tablets containing low water soluble drugs.

Authors' contributions

Conceptualization, methodology, T.G.M.; Investigation, G.P.; Visualization, S.H.M.K.; Visualization, M.S.M.D.; Draft preparation, G.C.; Checking grammatical errors and corrections, R.Y.

Acknowledgements

The authors are expressing their gratitude to Mr. B. Sudheer Chowdary participated in the work.

Funding

This study was not funded by any organization.

Availability of data and materials

All relevant data are within the paper and its supporting information files. Additional data will be made available on request according to the journal policy.

Conflicts of interest

There are no conflicts of interest.

References

- Kordi, M.; Farrokhi, N.; Pech Canul, MI.; Ahmadikhah, A.; Rice husk at a glance: From agro-industrial to modern applications. *Rice Sci.* 2024, 31(1), 14-32. <https://doi.org/10.1016/j.rsci.2023.08.005>
- Nawaz, S.; Jamil, F.; Akhter, P.; Hussain, M.; Jang, H.; Park, Y.K.; Valorization of lignocellulosic rice husk producing biosilica and biofuels—A review. *J. Phys.: Energy.* 2022, 5(1), 012003. <https://doi.org/10.1088/2515-7655/aca5b4> 10.1088/2515-7655/aca5b4
- Gao, Y.; Guo, X.; Liu, Y.; Fang, Z.; Zhang, M.; Zhang, R.; You, L.; Li, T.; Liu, R.H.; A full utilization of rice husk to evaluate phytochemical bioactivities and prepare cellulose nanocrystals. *Sci. Report.* 2018, 8(1), 10482. <https://doi.org/10.1038/s41598-018-27635-3>

4. Johar, N.; Ahmad, I.; Dufresne, A.; Extraction, preparation and characterization of cellulose fibres and nanocrystals from rice husk. *Industrial Crops and Products*. 2012, 37, 93-99. <https://doi.org/10.1016/j.indcrop.2011.12.016>
5. McMullen, R.L.; Ozkan, S.; Gillece, T.; Physicochemical properties of cellulose ethers. *Cosmetics*. 2022, 9(3), 52. <https://doi.org/10.3390/cosmetics9030052>
6. Deshpande, M.C.; Venkateswarlu, V.; Babu, R.K.; Trivedi, R.K.; Design and evaluation of oral bioadhesive controlled release formulations of miglitol, intended for prolonged inhibition of intestinal α -glucosidases and enhancement of plasma glucagon like peptide-1 levels. *International J. Pharm.* 2009, 3801-2, 1624. <https://doi.org/10.1016/j.ijpharm.2009.06.024>
7. Perioli, L.; Ambrogi, V.; Rubini, D.; Giovagnoli, S.; Ricci, M.; Blasi, P.; Rossi, C.; Novel mucoadhesive buccal formulation containing metronidazole for the treatment of periodontal disease. *J. Contr. Rel.* 2004, 953, 521-533. <https://doi.org/10.1016/j.jconrel.2003.12.018>
8. Ludwig, A. The use of mucoadhesive polymers in ocular drug delivery. *Adv. Drug Del. Rev.* 2005, 57, 1595-1639. <https://doi.org/10.1016/j.addr.2005.07.005>
9. Karasulu, Y.; Hilmioglu, H.; Metin, D. Y.; Güneri, T.; Efficacy of a new ketoconazole bioadhesive vaginal tablet on *Candida albicans*. *Il Farmaco*, 2004, 592, 163-167. <https://doi.org/10.1016/j.farmac.2003.11.018>
10. Sensoy, D.; Cevher, H.; Sarici, A.; Yilmaz, M.; Özdamar, A.; Bergisadi, N.; Bioadhesive sulfacetamide sodium microspheres: Evaluation of their effectiveness in the treatment of bacterial keratitis caused by *Staphylococcus aureus* and *Pseudomonas aeruginosa* in a rabbit model. *Eur. J. Pharm. Biopharm.* 2009, 723, 487-495. <https://doi.org/10.1016/j.ejpb.2009.02.006>
11. Barzegar-jalali, M.; Valizadeha, H.; Dastmalchi, S.; Siah Shadbad, MR.; Barzegar-Jalal, A.; Adibkia, K.; Mohammadi, G.; Enhancing dissolution rate of carbamazepine via cogrinding with crospovidone and hydroxy propyl methyl cellulose. *Iran. J. Pharm. Res.* 2007, 63, 159-165. <https://doi.org/10.22037/ijpr.2010.716>
12. Benchikh, L.; Merzouki, A.; Grohens, Y.; Guessoum, M.; Pillin, I.; Characterization of cellulose nanocrystals extracted from El Diss and El Retma local plants and their dispersion in poly (vinyl alcohol-co-ethylene) matrix in the presence of borax. *Polym. Polym. Composit.* 2021, (3),218-30. <https://doi.org/10.1177/09673911209108>
13. Rashid,S.; Dutta,H.; Physicochemical characterization of carboxymethyl cellulose from differently sized rice husks and application as cake additive. *LWT*. 2022 ,154,112630. <https://doi.org/10.1016/j.lwt.2021.112630>
14. Mora, C.F.; Kwan, A.K.; Chan, H.C.; Particle size distribution analysis of coarse aggregate using digital image processing. *Cement Concret. Res.* 1998, 28(6), 921-32. [https://doi.org/10.1016/S0008-8846\(98\)00043-X](https://doi.org/10.1016/S0008-8846(98)00043-X)
15. Buckman, Harry O.; Brady, Nyle C.; The Nature and Property of Soils - A College Text of Edaphology (6th ed.). New York City, Macmillan.1960, 50.
16. Pop, A.L.; Musuc, A.M.; Nicoară, A.C.; Ozon, E.A.; Crisan, S.; Penes, O.N.; Nasui, B.A.; Lupuliasa, D.; Secăreanu, A.A.; Optimization of the preformulation and formulation parameters in the development of new extended-release tablets containing felodipine. *Appl. Sci.* 2022, 12(11),5333. <https://doi.org/10.3390/app12115333>
17. Lowell, S.; Shields, J.E.; Powder surface area and porosity. Springer Science & Business Media, 2013.
18. Anthony, A.N.; Colin, M.M.; Vimal, J.P. Density determination of powders by liquid displacement methods. *Drug Dev. Ind. Pharm.* 1989, 15(4), 549-559. <https://doi.org/10.3109/03639048909040230>
19. Shah, R.B.; Tawakkul, M.A.; Khan, M.A.; Comparative evaluation of flow for pharmaceutical powders and granules. *Aaps Pharmscitech.* 2008,250-8. <https://doi.org/10.1208/s12249-008-9046-8>
20. Sarraguca, M.C.; Cruz, A.V.; Soares, S.O.; Amaral, H.R.; Costa, P.C.; Lopes, J.A.; Determination of flow properties of pharmaceutical powders by near infrared spectroscopy. *J. Pharm. Biomed. Anal.* 2010, 52(4), 484-92. <https://doi.org/10.1016/j.jpba.2010.01.038>
21. Deshmukh, H, Chandrashekhara, S.; Nagesh, C.; Patil, M.; Majethiya, V. Comparative evaluation of disintegrant in orodispersible tablet of aceclofenac. *Res. J. Pharm. Technol.* 2012, 5(6), 775-9.
22. Rashid, I.; Haddadin, R.R.; Alkafaween, A.A.; Alkaraki, R.N.; Alkasasbeh, R.M.; Understanding the implication of Kawakita model parameters using in-die force-displacement curve analysis for compacted and non-compacted API powders. *AAPS Open.* 2022, 8(1), 6. <https://doi.org/10.1186/s41120-022-00053-6>