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COMPOSITIONAL ANALYSIS AND YIELD OPTIMIZATION OF XYLAN EXTRACTION FROM RENEWABLE AGRO-INDUSTRIAL WASTE

Aiza Zafar[1], Mahwash Aziz[1]*, Rizwana Batool[1], Adan Naeem[1]

[1]Department of Food Science and Technology, Government College Women University, Faisalabad. Pakistan

***Corresponding author:** Mahwash Aziz
*Email: dr.mahwashaziz@gcwuf.edu.pk

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ABSTRACT

Agro-industrial waste is mostly composed of biomass derived from lignocellulose. Lignocellulose waste has been receiving great interest due to its readily available nature, mechanical and thermal capabilities, cost-effectiveness, low toxicity, biodegradability and renewable qualities. Corn husk is a renewable lignocellulosic biomass resource with significant potential for sustainable development and agricultural waste utilization. It contains cellulose, hemicellulose and lignin components. The main component of hemicellulose is xylan that can be used as a raw material or an intermediate in food and non-food industries. Current investigation is based on the yield optimization of xylan extraction from renewable agro-industrial waste after the determination of compositional analysis of corn husk that showed the high amount of organic, cellulose and hemicellulose content in it. Furthermore, extraction of xylan from corn husk was done by using steam as a pretreatment method that weakened the link between lignin, cellulose and hemicellulose making xylan available for extraction. After pretreatment, 12% NaOH was used for xylan extraction that produced high yield followed by the determination of reducing sugars (63 % xylose, 23 % arabinose and 14 % glucose) in its structure.

Keywords: Corn husk, agro-industrial waste, hemicellulose, xylan.

1. INTRODUCTION

The consumers are demanding sustainable and eco-friendly products due to high environmental concerns. In order to meet this demand, agricultural waste has been widely utilized in a variety of industries such as food, paper, composites, medical and textile as it prevents the buildup of unmanaged agro-waste. Using agricultural wastes for technical purposes also significantly lowers the danger of environmental damage. By-products of agricultural activities pertaining to crop or animal production are referred as agricultural wastes. They are composed of mostly lignocellulosic materials. Lignin, cellulose, and hemicellulose are among the main lignocellulosic materials found in agricultural waste (Rajinipriya *et al*., 2018).

Corn husk is a lignocellulosic substance that represents the leafy outer shell of maize ear as it grows on the plant. The most popular method of its disposal is to burn or bury them which is harmful for crops and cause pollution and resource waste. Components of maize husk include cellulose, hemicellulose and lignin. The cellulose microfibril bundles are surrounded by lignin and hemicellulose. Different factors such as the age of fiber, its source and the conditions under which it was extracted, all affect the percentage of cellulose, hemicellulose and lignin in the fiber (Ratna *et al*., 2022).

The hemicellulose content in cornhusk ranges from 30-45% including xylan as its main component. Xylan is made up of an aqueous xylose backbone (β-1,4-xylosyl residues) with a high degree of substitution. Additionally, xylan is joined to lignin and other structures by a ferulic acid bridge associated with arabinofuranose group on the backbone. The benefits of xylan in the food and non-food industries are widely recognized with distinctive features. It has the potential to replace sucrose in the food industry due to its slow digestion, low calorie content and sweetness. Furthermore, it improves human health by stimulating the growth of bifidobacteria in colon. Xylan is also being used in the creation of edible films and coatings of medicines (Agustinisari *et al*., 2024).

Initially, xylan is extracted from different agricultural byproducts using an alkali or combination of alkaline solutions. However, the amount of xylan recovered from plant materials using alkaline extraction depend on number of factors such as composition of plant material, type and concentration of alkali being used, extraction conditions along with the amount of lignin present and its bonding with the matrix of plant cell wall. In order to extract xylan, a number of pretreatments that target either the lignin fraction or cellulose have been proposed to weaken the lignin cellulosic linkages (Samanta *et al*., 2012). Current study is based on the compositional analysis of corn husk followed by extraction of xylan by using steam application as pretreatment followed by NaOH hydrolysis to increase its recovery from corn husk.

2. PROCUREMENT OF RAW MATERIALS

Corn husk of freshly harvested corns was collected from Ayub Agricultural Research Institute (AARI) Faisalabad. The chemicals and standards used were purchased from Sigma Aldrich (Sigma Aldrich, Tokyo, Japan) and Merck (KGaA Merck Darmstadt, Germany).

2.1. Preparation of Corn Husk Powder

Corn husk powder was prepared by following the method as described by Samanta *et al.* (2016).

2.2. Chemical Characterization of Corn Husk Powder

2.2.1. Moisture and Ash Content

Total Moisture and ash content of corn husk powder were determined by following the method of AACC (2019).

2.2.2. Organic Content

Organic content of corn husk powder was determined by following the method of Samanta *et al.* (2016).

2.2.3. Cellulose, Hemicellulose and Lignin

The amount of lignin, cellulose and hemicellulose in corn husk powder was determined by following the method of Pereira *et al.* (2022) with some modifications. 2g sample extraction was done in duplicates with ether in Soxhlet assembly for 6 h. After that these defatted samples were boiled with 0.5% ammonium oxalate in the ratio of 1:50 to remove water soluble matter and pectin. Then bleaching of sample was done with 1% solution of sodium chlorite and 0.05 N acetic acid in boiling water bath for 1h to remove lignin. Initially, hemicellulose was removed by treating holocellulose with 5% (for 2 h) and 24% (for another 2 h) potassium hydroxide. Furthermore, cellulose was obtained by using 90% phosphoric acid with 20-fold volume and stirred for 2 h at 1 °C followed by washing with 1% sodium carbonate solution and water. The resulting residue was dried in incubator at 37°C and weighed till constant weight was obtained. The ash content was then subtracted from residual weight to obtain the actual pure cellulose weight.

2.3. Extraction of Xylan

According to the method of Yan *et al.* (2022), xylan was extracted with the help of sodium hydroxide as it is highly efficient to hydrolyse ester linkages in corn husk followed by aqueous medium extraction. 12% sodium hydroxide was used followed by steam application at 121℃ for 45 min under 15 lb pressure. The extract was then centrifuged at 5000 rpm for 20 min followed by neutralization at pH 5 with glacial acetic acid. Furthermore, cold ethanol (3 volumes) was used to precipitate the resulted neutralized extract. This precipitated xylan was then centrifuged at 8000 rpm for 10 min at 4 ℃. After centrifugation the resultant xylan was then dried in hot air oven at 60-70 ℃ till constant weight was obtained. The obtained dried pallets were then weighed and transformed into powder by using a mixer.

2.4. Yield of Xylan

The yield of xylan was determined according to the method of Khat-Udomkiri *et al.* (2018) by using following formula:

$$\text{Xylan Yield (\%)} = \frac{\text{Dry weight of extracted xylan (g)}}{\text{Weight of the sample (g)}} \times 100$$

2.5. Quantification of Reducing Sugars

Quantification of reducing sugars in extracted xylan was done by following the method of Samanta *et al.* (2016). The GOD/POD kit was used to estimate glucose in corn husk xylan. After the hydrolysis with sulphuric acid, xylan was examined using HPLC. In order to eliminate extraneous materials, acid hydrolysate was run through a 0.45µm syringe filter. The sample was then analyzed by HPLC equipped with refractive index detector (RID) using Zorbex carbohydrate analysis column. Manual injector was used to load 20 µl of filtered material after the column temperature was set at 25°C. A solvent mixture of acetonitrile and water was used to elute the sugar monomers at a flow rate of 0.5 ml/min. The concentration of each sugar was measured and calculated as percentage by using average peak areas in comparison to the mixture of standard sugars (xylose, glucose, and arabinose).

2.6. Statistical Analysis

The data is presented as means ± standard deviations (SD). The mean and SD for each variable were calculated. All analyses were conducted using Statistix 8.1 software.

3. RESULTS AND DISCUSSION

3.1. Corn husk powder

Resultant corn husk powder was in dried form and obtained after drying husks in hot air oven at 70 ± 2 ℃ temperature till constant weight. Afterwards, dried corn husk was grind uniformly and passed through sieve of 20 mesh size to attain fine corn husk powder (Anh and Tai, 2024).

3.2. Physical and Chemical Characterization of Corn Husk Powder

3.2.1. Moisture Content

The amount of moisture in maize husk might change depending on the drying process and ambient conditions. The moisture content of corn husk powder is represented in Table 1. Stabilizing moisture content of corn husk is crucial for xylan extraction to prevent spoilage and for producing accurate processing outcomes. Moisture level should be kept to a minimum while extracting xylan from corn husk as the high moisture content will cause the corn husk powder to clump together and affect the extraction process. In physical assessment of corn husk, Mendes *et al*. (2015) found that the range of moisture content for various samples of corn husk was 7.6-8.7. Kambli *et al*. (2017) assessed the physiochemical characteristics of maize husk and also found that it contained 11.4% moisture content. According to a study by Mayta *et al*. (2024), the moisture content of maize husk was 11.2% which is near to the ideal range.

3.2.2. Ash Content

Ash is the inorganic residue that remains after organic matter has been removed as determined by compositional analysis. This ash is primarily composed of minerals such as calcium, magnesium, silica and potassium with trace amounts of other components. Ash content of corn husk powder is represented in Table 1. Minerals like potassium, silica, magnesium and calcium mainly make up this ash with trace amount of other elements. According to a compositional analysis of maize husks by Ibrahim *et al*. (2020), 0.36% ash concentration was observed. Similarly, the results are in accordance with Samanta *et al*. (2016) who conducted a compositional analysis on maize husk and found that 2.89% of the husk was ash.

3.2.3. Organic content

The determined organic content in corn husk powder is represented in Table 1. Higher amounts of cellulose, hemicellulose and lignin were found along with the trace amounts of ash, protein, enzymes, lipids and phenolic substances.

3.2.4. Cellulose

Cellulose is one of the major components of plant cell walls which gives corn husk its structural stability. The analyzed cellulose content of corn husk powder is given in Table 1. The results obtained are consistent with those of Mendes *et al*. (2015) who characterized maize husk and found that several samples contained 31.3 to 39.3% cellulose. Another study by Enawgaw *et al*. (2023) found that the cellulose content of maize husk ranged from 21.93 to 44.5%. Similarly, Ibrahim *et al*. (2020) examined the chemical makeup of maize husk and revealed a cellulose level of 45.7%. Additionally, Anh and Tai (2024) assessed the chemical makeup of corn husk powder and found that the sample contained 35.90% cellulose.

3.2.5. Hemicellulose

The main component of maize husk powder that contributes to its structural qualities is hemicellulose. Typically, it is made up of a combination of polysaccharides primarily xylan with smaller amounts of glucose, mannose and arabinose. The findings are consistent with those of Enawgaw *et al*. (2023), who examined the range of 21.93-44.5% hemicellulose in maize husk. The characterization of maize husk was also examined by Mendes *et al.* (2015) who found that 34.0-41.0% of hemicellulose was present in various corn husk samples. Furthermore, Ibrahim *et al.* (2020) and Anh and Tai (2024) determined the hemicellulose concentration by evaluating the chemical composition of corn husk to be 35.8 and 30.23%, respectively.

3.2.6. Lignin

The rigidity and structural stability of maize husk cell walls are attributed to the complex polymer of lignin. The presence of lignin along with cellulose and hemicellulose gives maize husks their rigid, complexed and cross-linked structure along with fibrous texture. To break down or get rid of lignin, pretreatment methods including oxidative, acidic or alkaline treatments are usually needed. These pretreatments improve xylan availability and extraction by reducing lignin levels and loosening the cell wall structure. Phenolic compounds and other byproducts may be released during the pretreatment process as lignin breaks down. These could hinder extraction and xylan quality if they are not sufficiently managed or removed. Breakdown of lignin during pretreatment may release byproducts such as phenolic compounds. If these are not adequately controlled or eliminated, they may prevent extraction and quality of xylan. Therefore, it is important to control these chemicals by utilizing pretreatment techniques such as ion-exchange resins, activated carbon and other adsorbents as well as by controlling pretreatment variables like temperature, time duration and pH (Khan *et al*., 2022). The amount of lignin in corn husk powder is represented in Table 1. The results are consistent with the work conducted by Mendes *et al.* (2015), who reported 7.97% lignin in maize husk. Similarly, Anh and Tai (2024) conducted experiments on the chemical composition of maize husk powder and found that the lignin percentage was 13.41%.

Table 1. Physical and chemical characterization of corn husk powder

Content	Corn Husk Powder (CHP) %
Moisture	8.74±0.05
Ash	4.50±0.06
Organic content	96.21±0.11
Cellulose	41.25±0.14
Hemicellulose	35.55±0.12
Lignin	6.25±0.08

3.3. Extraction of Xylan

Sodium hydroxide effectively breaks down hemicellulose to release xylan when steam is applied. This technique is frequently employed to extract xylan from lignocellulosic biomass. It is a more cost-effective method of xylan extraction. NaOH breaks ether and ester bonds in lignin, hemicellulose and cellulose matrix while degrading the structure of lignin in the process. In present study, steam was used as pretreatment technology to increase xylan yield. Moreover, the concentration of NaOH was 12% which caused a considerable proportion of lignin to dissolve successfully. This treatment encouraged the release of xylan that contained hemicellulose by reducing the amount of lignin. As corn husk fibres swelled more easily in an alkaline environment, hemicellulose having xylan was more accessible. According to Kumari *et al*. (2024), alkaline pretreatment with 12% NaOH can increase xylan recovery to as much as 70 to 90% depending on the processing parameters. Results of this study showed that corn husk powder treated with 12% alkali after steam application caused high xylan yield. Since cellulose is insoluble in sodium hydroxide and lignin is not precipitated by alcohol, the sample that was recovered following alkaline extraction was appeared to be xylan.

3.4. Yield of Xylan

Depending on specific parameters like temperature, pressure, alkali content and reaction time, the yield of xylan obtained by steam and NaOH extraction may vary significantly. In particular, the steam extraction method and NaOH were commonly used for maize husks because they yield the maximum amount of partially hydrolysed xylan with the highest purity. The corn husk xylan yield is presented in Table 2. The findings are in consistent with those of Samanta *et al*. (2016), who used 12% NaOH in conjunction with steam application to produce high xylan yield from corn husk. Similarly, Khat-Udomkiri *et al*. (2018) stated that the maximum yield of xylan was obtained by optimizing the alkaline pretreatment of rice husk.

Table 2. Yield of corn husk xylan

Sample	Yield (%)
Corn husk xylan	76.10±0.12

3.5. Quantification of Reducing Sugars

The hydrolysis of xylan caused reduction in its sugar content. With smaller amounts of arabinose, xylose is the main monomer in the reducing sugar content of xylan made from corn husks. Figure 1. displays the amount of xylose, arabinose and glucose that HPLC analysis found in the xylan of corn husk.

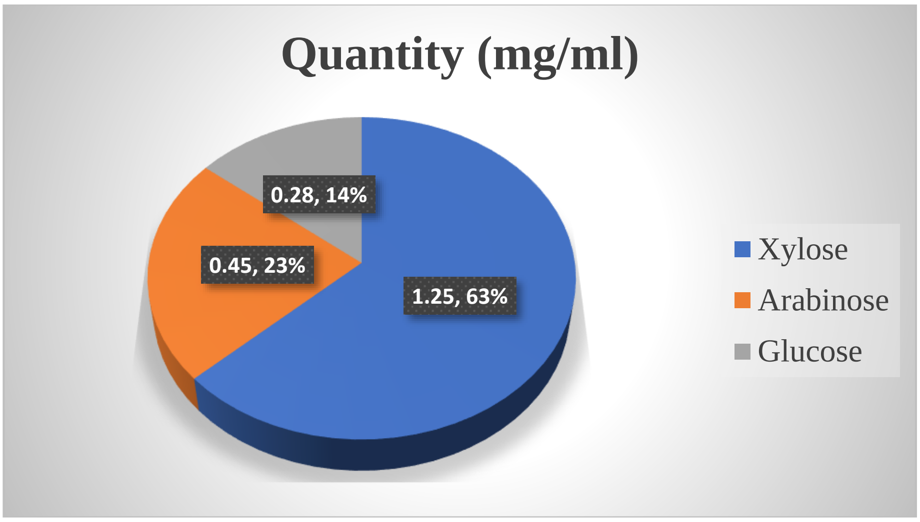

Fig 1. Amount of reducing sugars in xylan

4. CONCLUSION

Current study results showed that corn husk is an agro-industrial waste that can be used successfully to obtain high amount of hemicellulose containing xylan. The steam as pretreatment method disrupted lignocellulosic structure increasing the accessibility of hemicellulose. Significantly an effective xylan solubilization and recovery was made possible by subsequent alkaline extraction by using 12% sodium hydroxide. The method supports the utilization of agricultural waste such as corn husk into valuable biopolymers. The xylan recovered can further be used to produce xylooligosaccharides as functional ingredient to create edible films and biodegradable packaging and as fat replacer along with thickening and stabilizing agent in food products.

REFERENCES

1. Agustinisari, I., H. Herawati, N. Harimurti, E. Hermiati and I. Zahrina. 2024. The Properties of Xylan Extracted from Corncob Using Deep Eutectic Solvent. IOP Conference Series: Earth and Environmental Science, IOP Publishing.
2. American Association of Cereal Chemists (AACC). 2019. Approved methods of the Analysis. 11th Ed. Cereals & Grains Association, St. Paul, MN, U.S.A.
3. Anh, P. T. H. and D. M. Tai. 2024. Closer Approach towards the Preparation of Cellulose and Microcrystalline Cellulose from Corn Husks. Chemical Engineering & Technology. 47(9): 202300379.
4. Enawgaw, H., T. Tesfaye, K. T. Yilma and D. Y. Limeneh. 2023. Multiple Utilization Ways of Corn By-Products for Biomaterial Production with Bio-Refinery Concept; a Review. Materials Circular Economy. 5(1): 7-20.
5. Ibrahim, M., S. Sapuan, E. Zainudin and M. Zuhri. 2020. Preparation and characterization of cornhusk/sugar palm fiber reinforced Cornstarch-based hybrid composites. Journal of Materials Research and Technology. 9(1): 200-211.
6. Kambli, N. D., V. Mageshwaran, P. G. Patil, S. Saxena and R. R. Deshmukh. 2017. Synthesis and characterization of microcrystalline cellulose powder from corn husk fibres using bio-chemical route. Cellulose. 24: 5355-5369.
7. Khat-Udomkiri, N., B. S. Sivamaruthi, S. Sirilun, N. Lailerd, S. Peerajan and C. Chaiyasut. 2018. Optimization of alkaline pretreatment and enzymatic hydrolysis for the extraction of xylooligosaccharide from rice husk. AMB Express. 8: 1-10.
8. Kumari, K., S. Nagar, S. Goyal, S. Maan, V. Kumar, N. Kharor, M. Sindhu and V. Kumar. 2024. A fast, reliable, low-cost, and efficient xylan extraction for xylooligosaccharides production. Biofuels, Bioproducts and Biorefining. 18(5): 1355-1368.

9. Mayta, S., R. G. Huamani-Palomino, B. M. Córdova, E. Rivera and M. Quintana. 2024. Utilizing peracetic acid as an eco-friendly bleaching agent: investigating whiteness levels of cellulose microfibers from corn husk waste. Biomass Conversion and Biorefinery. 1-13.
10. Mendes, C., F. Adnet, M. Leite, C. G. Furtado and A. Sousa. 2015. Chemical, physical, mechanical, thermal and morphological characterization of corn husk residue. Cellul. Chem. Technol. 49: 727-735.
11. Pereira, B. S., C. de Freitas, J. Contiero and M. Brienzo. 2022. Enzymatic production of xylooligosaccharides from xylan solubilized from food and agroindustrial waste. BioEnergy Research. 1-9.
12. Rajinipriya, M., M. Nagalakshmaiah, M. Robert and S. Elkoun. 2018. Importance of agricultural and industrial waste in the field of nanocellulose and recent industrial developments of wood based nanocellulose: a review. ACS Sustainable Chemistry & Engineering. 6(3): 2807-2828.
13. Ratna, A. S., A. Ghosh and S. Mukhopadhyay. 2022. Advances and prospects of corn husk as a sustainable material in composites and other technical applications. Journal of Cleaner Production. 371: 133563-133591.
14. Samanta, A. K., A. Kolte, A. Elangovan, A. Dhali, S. Senani, M. Sridhar, K. Suresh, N. Jayapal, C. Jayaram and S. Roy. 2016. Value addition of corn husks through enzymatic production of xylooligosaccharides. Brazilian Archives of Biology and Technology. 59: 16160078-16160085.
15. Samanta, A., S. Senani, A. P. Kolte, M. Sridhar, K. Sampath, N. Jayapal and A. Devi. 2012. Production and in vitro evaluation of xylooligosaccharides generated from corn cobs. Food and Bioproducts Processing. 90(3): 466-474.
16. Yan, F., S. Tian, K. Du, X. a. Xue, P. Gao and Z. Chen. 2022. Preparation and nutritional properties of xylooligosaccharide from agricultural and forestry byproducts: A comprehensive review. Frontiers in Nutrition. 9: 977548-977567.