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Investigating the characteristics of lignin, carboxymethyl cellulose and starch extracted from residues of corn and

Razieh Nimazam[1*]; Parvin Sharayee [2*]; Vahideh Sharif[3]; Mahboobeh Sarabi Jamab[4]; Soudabeh Ain Afshar[2]; Elham Azarpajouh[2]; Farzaneh Sharif[3]

1-Food Chemistry Department, Food Science and Technology Research Institute, Mashhad, Iran.
2-Agricultural Engineering and Technical Research Department, Agricultural and Natural Resources Research and Education Center, Khorasan Razavi Province, Agricultural Research, Education and Extension Organization, Mashhad, Iran.
3-Saffron Research Institute, Torbat Heydariyeh University, Torbat Heydariyeh, Iran.
4-Food Biotechnology Department, Food Science and Technology Research Institute, Mashhad, Iran

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*Corresponding Author E-

r.niazmand@rifst.ac.ir

parvin_sharayei@yahoo.com

ABSTRACT

This research aimed to produce high value-added products, including starch and carboxymethyl cellulose from saffron corms and corncob waste respectively, as well as active extracts from saffron corms and lignin from corncob residues. In this study, the extraction yield, antioxidant capacity, and antifungal properties of extracts obtained from saffron corms and lignin produced from corncob waste, along with the physicochemical properties of starch derived from saffron corms and carboxymethyl cellulose produced from corncob residues were investigated. The extraction yields of saffron corm extract and lignin from corn residues were more than 14% and 17%, respectively. The phenolic content and radical scavenging activity of the saffron corm extract were 137.91 mg per 100 g dry weight and 29.31%, while for corn lignin, they were 156.69 mg per 100 g dry weight and 58.85%, respectively. The saffron corm extract and lignin exhibited antifungal activity but the minimal inhibitory concentration (MIC) and minimal fungicidal concentration (MFC) of lignin were lower than those of the saffron corm extract due to the higher content of phenolic compounds in lignin. The ash and protein content of starch obtained from saffron onion was 0.16 and 0.75%, respectively, which is close to potato starch. The extraction yield of cellulose from corn wood residues was 38.43%, and the purity grade and degree of substitution of the carboxymethyl cellulose prepared from it were 73% and 85%, respectively. The results obtained from Fourier-transform infrared spectroscopy (FTIR) confirmed the formation of carboxymethyl cellulose from cellulose extracted from corn wood. According to the results of the present study, it can be said that food waste and residues can be used as sustainable and low-cost resources for the production of valuable products such as cellulose and starch with industrial applications.

1. Introduction

Reducing agricultural waste not only returns a significant volume of products to the consumption cycle but also enables substantial progress toward increasing the productivity of production resources, including water and other production inputs such as soil, fertilizer, pesticides, fuel, and energy. This has drawn the attention of the global community and researchers toward the optimal utilization of waste and by-products from the agricultural and food industries. Among the extensive agricultural wastes, the waste derived from saffron corms holds particular importance in Iran.

Iran produces and exports approximately 90 to 93 percent of the world's saffron [1]. Saffron grows well in arid and semi-arid regions that experience cold winters and hot, dry summers. According to the Ministry of Agriculture Jihad statistics, saffron production in 2022 was approximately 377 tons, of which about 290 tons were produced in Razavi Khorasan Province, 54 tons in South Khorasan, and approximately 12 tons in North Khorasan. Specifically, for every 1 kilogram of saffron spice produced, approximately 63 kilograms of flower biological residues are generated, including about 53 kilograms of petals, 9 kilograms of stamen, and 1 kilogram of style, along with 1500 kilograms of leaves and 100 kilograms of corms [2]. The corm is the underground stem of the saffron plant that is responsible for plant propagation. Saffron corms contain valuable compounds including phenolic compounds, glucose, starch, amino acids, fatty acids, sterols, and saponins [3].

Corn is another highly valuable and practical crop in the country. The cultivation of this plant has received attention for several decades with increasing focus on livestock and poultry activities, and after the positive reception from livestock farmers, broiler and laying hen producers, and industrial and pharmaceutical factories, it has been seriously pursued. Corn grain, which belongs to the important cereal family, possesses plant organs including root, stem, leaf, flower, and seed. The product of this plant, called the cob, is harvested for nutritional utilization purposes. Considering the 52 percent harvest coefficient of corn grain [4] and the production of 1,101 thousand tons of corn grain in 2019 [5], the amount of corn grain plant residues is estimated to be approximately 572 thousand tons. Corn grain plant residues consist of 50% stem, 20% leaf, 20% cob wood, and 10% husk and sheath [6]. Currently, corn grain waste has not received much attention and is often regarded as waste material. Not only is optimal use not made of these materials, but in some cases these residues are disposed of or burned through improper methods, which will have adverse effects on the environment. However, these residues are rich in nutrients and highly valuable chemical compounds such as cellulose and lignin. Perumal et al. (2022) discussed the extraction of cellulose nanocrystals (CNCs) from areca nut waste and their potential application in biocomposite films. This study demonstrated that the resulting nanobiocomposite films showed good antimicrobial activity against foodborne pathogens and postharvest fungi, indicating their suitability for food packaging applications [7]. In another study, two pulping and chemo-mechanical methods for extracting cellulose fiber from rice straw for potential use as a biodegradable reinforcement in polymeric materials were investigated [8]. Fronza et al. (2023) evaluated the economic and technical aspects of starch extraction from avocado seeds, which are typically considered waste. They stated that the extraction process is technically feasible and yields considerable starch that can be used in various industries, including food and bioplastics [9].

With these explanations, the objective of this research was to focus on the waste and residues of these two valuable products. In this study, the production of high value-added products with high commercial applications, including starch and carboxymethyl cellulose from saffron corm waste and corn residues respectively, was investigated. Additionally, the possibility of producing active extracts from them, including saffron corm extract and corn lignin, was studied, and their antioxidant and antimicrobial properties were examined.

2. Materials and Methods

2.1. Materials

The materials used in this research were purchased from Sigma and Merck companies. Aspergillus flavus ATCC and Aspergillus parasiticus ATCC molds were purchased from the microbial collection of the Industrial Research Organization of Iran. Microbial culture media including Potato Dextrose Broth (PDB) and Potato Dextrose Agar (PDA) as well as Ringer tablets were purchased from Merck Germany.

2.2. Preparation of Saffron Corm

Saffron corms were randomly harvested during dormancy from one of the farms in Khorasan Razavi province in Zaveh county and transferred to the laboratory. First, the outer husks around the corms were removed, then they were washed with distilled water, cut into small pieces, and dried in an oven at 50 degrees Celsius. Finally, the dried sample was converted to fine powder using a mill (Decosab-Iran) and passed through a sieve (Damavand-Iran) with 35 mesh and used for subsequent tests.

2.2.1. Physicochemical Tests of Saffron Corm Powder

2.2.1.1. General Composition

The composition of saffron corm powder was determined based on standard AOAC (2005) methods [10]. Moisture percentage determination was performed through drying using an electric oven. Ash was determined using an electric furnace (Ex.1200.2L, Excition Co., made in Iran) at a temperature of 550 degrees Celsius. Protein measurement was performed using a Kjeldahl apparatus (Gerhardt, made in Germany) with a conversion factor of 6.25, and crude fiber was determined by the Weende method using a fiber analyzer. Fat determination was performed using a Soxhlet apparatus. The total carbohydrate content was also calculated by subtracting the sum of moisture, protein, ash, fiber, and fat from one hundred.

2.2.2. Extraction from Saffron Corm

Extract extraction was performed using the immersion method in 80 percent ethanol solvent (for 48 hours) (Figure 1). After completion of the process, the solution was filtered under vacuum and concentrated using a rotary evaporator under vacuum (model Laborota 4000 efficient, made in Germany) at 45 degrees Celsius until complete dehydration. To prevent foaming and also to reach the appropriate temperature for entering the freeze dryer, the concentrated solution was kept in a freezer at -70 degrees Celsius for 19 hours and then transferred to a freeze dryer (model Operon FDB-550, made in Korea). The samples (approximately 200 milliliters) were dried in the freeze dryer at -55 degrees Celsius with a pressure of 0.15 mmHg for 20 hours. The dried samples were stored in darkness at -18 degrees Celsius until subsequent tests were performed.

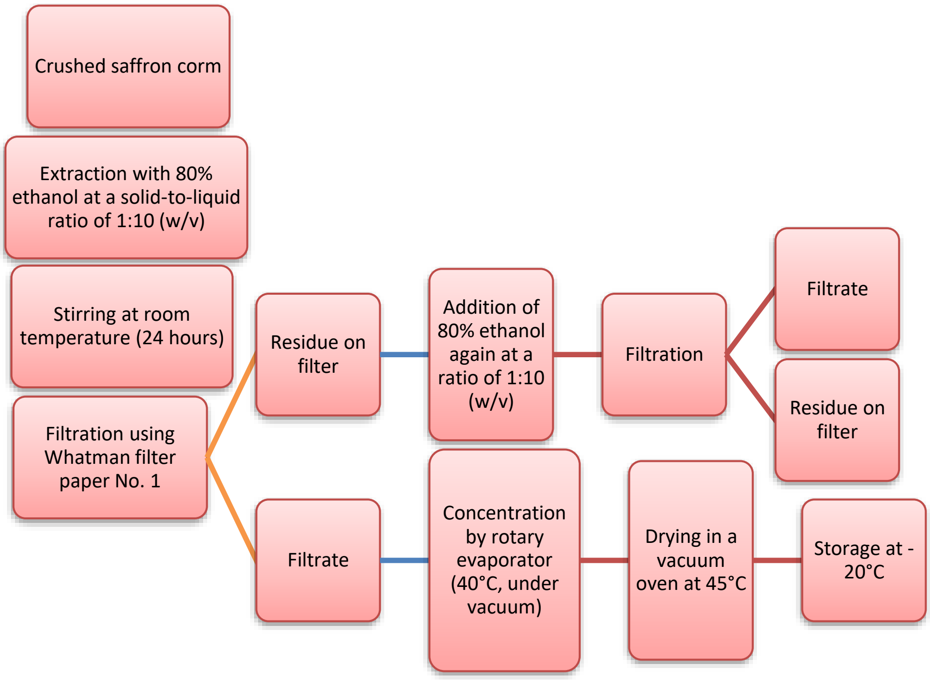


Fig 1: Extraction of saffron corm extract by immersion method

2.2.3. Determination of Total Extraction Yield

To determine the extraction yield, the freeze-dried extract obtained from extraction was carefully weighed. Finally, considering the initial ratio of 1:10 corm powder to solvent, the total yield was reported as grams of extract per 100 grams of dry saffron corm [11].

2.2.4. Determination of Total Phenolic Compounds

To determine total phenolic content, 0.5 mL of extract was added to 2.5 mL of Folin-Ciocalteu reagent (at 10 percent v/v concentration). After 4 minutes, 2 mL of 7.5 percent sodium carbonate was added. The absorption of the mixture was measured after 30 minutes of incubation at room temperature at 765 nanometers wavelength using a UV-visible spectrophotometer. Gallic acid was used as the standard, and results were expressed as milligrams of gallic acid equivalent per 100 grams of dry corm powder [12].

2.2.5. Evaluation of Antioxidant Capacity by DPPH Free Radical Scavenging Power Assay

To determine antioxidant capacity using the DPPH method, 1 mL of corm extract was mixed with 2 mL of methanolic DPPH reagent solution (at 0.004 percent w/v concentration). The mixture was shaken and kept at room temperature for 30 minutes. Then its absorption was measured at 517 nanometers wavelength against pure solvent. The DPPH solution was prepared daily and stored in a glass flask covered with aluminum foil. The antioxidant capacity of samples was calculated as the percentage of DPPH radical inhibition according to equation 1 [13].

$$\text{Inhibition } (\%) = 100 \times \left(\frac{A_{blank} - A_{sample}}{A_{blank}}\right) \quad [1]$$

where A_{blank} was the control absorption and A_{sample} was the sample absorption.

2.2.6. Ferric Reducing Antioxidant Power (FRAP) Assay

The reagent used in the iron reduction-based antioxidant power determination method is a mixture of sodium acetate buffer (300 mmol per liter, pH 3.6), 2,4,6-tris(2-pyridyl)-s-triazine solution (10 mmolar prepared in 40 mmolar hydrochloric acid), 20 mmolar iron (III) chloride solution, and distilled water in the ratio of 2:1:1:1.10, respectively, which is prepared daily. To measure antioxidant properties, 0.2 mL of sample was added to 1.8 mL of FRAP reagent. The absorption of the mixture was measured at 593 nanometers wavelength after 10 minutes of incubation at 37 degrees Celsius. The standard curve was drawn using iron (II) sulfate solution, and results were calculated as millimoles per liter of iron (II) per 100 grams of corm powder. Dilution was performed for samples when necessary [14].

2.3. Investigation of Antibacterial Properties of Saffron Corm Extract and Lignin Derived from Corncob

2.3.1. Activation of Microorganisms

To prepare fungal suspension, subcultures were first prepared from the primary cultures of Aspergillus flavus and Aspergillus parasiticus using a sterile loop and placed in an incubator at 25 degrees Celsius so that after 5 days the molds would enter the logarithmic phase and sporulate. The spores of the fungi cultured on potato dextrose agar medium were scraped sterilely and transferred into sterile Ringer's solution containing Tween 80, and the number of fungal spores was counted using a hemocytometer slide. Finally, the number of spores of Aspergillus parasiticus and Aspergillus flavus were counted as 1.1×10^6 and 1.4×10^6 colony-forming units (CFU) per milliliter, respectively.

2.3.2. Measurement of Microbial Growth Inhibition Zone

The disk diffusion method was used to perform this test. To conduct the test, microbial suspensions with concentrations equivalent to 10^6 and 10^3 colony-forming units per milliliter of Aspergillus flavus and Aspergillus parasiticus spores were first prepared. Then, 100 microliters of microbial suspensions were uniformly spread on the surface of potato dextrose agar medium. Sterile blank disks with a diameter of 6 millimeters were impregnated with 30 microliters of saffron corm extract at a concentration of 0.5 mg per liter and also with 30 microliters of lignin at a concentration of 0.05 mg per liter, and then placed on the agar medium surface. It should be noted that the extract was passed through a 0.45 micrometer sterile filter before use. Disks soaked with water and dimethyl sulfoxide (DMSO) were placed on the agar medium surface as negative controls, and disks soaked with 200 mg per liter ketoconazole solution were placed as positive controls. The plates were incubated at 25 degrees Celsius for 72 hours, and then the diameter of growth inhibition zones around the disks was measured and reported in millimeters [15].

2.3.3. Determination of Minimum Inhibitory Concentration (MIC) and Minimum Fungicidal Concentration (MFC) by Microdilution Method

To determine the minimum inhibitory concentration of fungal growth for the samples, the microdilution method was used employing turbidometry with an ELISA reader (ELX 808, Biotech, made in USA). This was done by first adding 100 microliters of different dilutions of saffron corm extract and lignin (500, 250, 125, 62.5, 31.25, 15.625, 7.8125, 3.925, 0.19 mg per milliliter) to microtubes containing 100 microliters of PDB medium and 10 microliters of microbial suspension (1.1×10^6 colony-forming units per milliliter). Wells containing culture medium and antimicrobial compounds without microbial suspension were prepared as negative controls, and wells containing liquid culture medium and 10 microliters of microbial suspension without antimicrobial compounds were prepared as positive controls. The ELISA plate was then incubated at 25 degrees Celsius. Microbial growth was determined by measuring optical density using the ELISA device. The minimum growth inhibitory concentration was defined as the lowest concentration of saffron corm extract or lignin with turbidity similar to negative control wells as the minimum fungal growth inhibitory concentration. To determine the minimum fungicidal concentration of antimicrobial compounds, 100 microliters from wells with turbidity similar to or less than the negative control were spread on PDA culture medium surface and incubated at appropriate temperature and time for microorganisms. The minimum fungicidal concentration is actually the lowest concentration of antimicrobial compounds that causes death of microorganisms such that no colonies are observed on the plates [16].

2.4. Extraction and Purification of Starch

Saffron corm powder was soaked for 2 hours in water containing sodium metabisulfite (0.075 percent) at a ratio of 1:2 (w/v) [17]. After 10 minutes of centrifugation (2000 rpm), water was added at four times the volume, and the pH was made alkaline to 8 using 1 molar sodium hydroxide/hydrochloric acid solutions. The suspension was stirred for 30 minutes at room temperature using a magnetic stirrer to dissolve existing proteins and facilitate starch separation [18]. The mixture was kept motionless for 2 hours to allow starch to settle. After removing the floating layer, the settled layer was mixed with a little water and centrifuged for 10 minutes at 2000 rpm. The mixture of the settled layer with water was passed through a sieve with 200 mesh. To remove existing fats and proteins, the starch sediments were stirred overnight in salt water with 10 degrees Brix along with toluene (one-eighth volume) [17]. Then the toluene layer was removed by centrifugation and the gray layer on the surface of sediments was separated using a spoon. The settled layer was washed once with water, once with acetone, and 5 times with distilled water. Finally, the purified starch was dried for 24 hours in an oven at 35 degrees Celsius. The obtained starch was gently crushed and passed through a sieve with 100 mesh. The starch extraction yield was calculated using equation 2.

$$\text{Starch yield } (\%) = 100 \times \left(\frac{S}{Sp}\right) \qquad [2]$$

where S is the amount of extracted starch (grams) and SP is the initial amount of saffron corm powder (grams).

2.4.1. Physicochemical Characteristics of Saffron Corm Starch

2.4.1.1. General Composition

Starch samples were examined for crude protein content (N×6.25), fat content, and ash content [10].

To measure amylose content, the color of the iodine-starch complex obtained at pH around 4.5 to 4.8 in acetate buffer was read at 620 nanometers wavelength using a UV-visible spectrophotometer. A standard curve of amylose solution in water with concentrations of 0.004 to 0.024 mg per milliliter was used to measure amylose concentration in the sample [19].

2.4.1.2. Determination of Color Components

Approximately 500 mg of sample was placed between two glass slides and its image was captured using a scanner model (Hp Scan Jet G3010). To prevent any lateral light entry, the scanner surface was covered with completely black and thick fabric. Images were saved at 300 dpi resolution in JPEG format. Following transfer of the images to the computer, their color coordinates were extracted in the Lab color space using ImageJ software (version 1.40g). The Lab color model consists of the L component (lightness) with a range from zero (black) to 100 (white), the a component (redness) which is unlimited with a color spectrum ranging from green (negative values) to red (positive values), and the b component (yellowness) which is unlimited with a color spectrum ranging from blue (negative values) to yellow (positive values) [20].

2.5. Extraction of Compounds from Corn Waste

2.5.1. Lignin

Lignin extraction was performed using the acidic dioxane method with minor modifications and adjustments [21]. For this purpose, corn wood was first chopped into small pieces and then converted to fine powder using a mill (Dekosab, made in Iran) and passed through a sieve (Damavand, made in Iran) with 60 mesh. To separate extractive materials and carbohydrates, the resulting powder was washed with acetone and then with water, each time for 6 hours in a Soxhlet extractor. Twenty-five grams of the powder free from extractive materials, along with 90 milliliters of dioxane and 10 milliliters of 37 percent hydrochloric acid, was heated for 10 hours in a hot water bath at 67 degrees Celsius. Subsequently, the dioxane containing lignin and acid was filtered through filter paper and precipitated in distilled water. The resulting lignin was washed with distilled water until complete acid removal and dried at ambient temperature. The lignin extraction yield was calculated using equation 3.

$$\text{Lignin yield } (\%) = 100 \times \left(\frac{l}{lp}\right) \qquad [3]$$

where l is the amount of extracted lignin (grams) and lP is the initial amount of corn wood (grams).

2.5.2. Extraction of Cellulose from Corn Wood

For this purpose, corn wood was first chopped into small pieces and then converted to fine powder using a mill (Dekosab, made in Iran) and passed through a sieve (Damavand, made in Iran) with 60 mesh. Subsequently, corn wood powder and water were mixed at a water-to-pulp ratio of 20:1 in a mixer for 10 minutes. This process was performed to remove fleshy materials adhered to the corn wood husk. The mixture was passed through a 250-micrometer filter and washed several times with distilled water. Corn wood husk waste contains starch, cellulose, hemicellulose, lignin, and other impurities [22] and requires several stages of alkaline pretreatments and bleaching prior to acid hydrolysis to separate cellulosic compounds [23]. The compounds prepared from the above stage were stirred with 0.5 normal sodium hydroxide solution at 80 degrees Celsius for 2.5 hours. This stage was repeated three times to completely remove lignin, hemicelluloses, and other impurities. After each process, the pulp was washed with distilled water and passed through a 75-micrometer filter to remove alkaline solutions and undissolved impurities. Subsequently, bleaching was performed using 2.3 percent by weight sodium chlorite solution in acetate buffer (pH=9). The purpose of bleaching is to remove organic residues. The bleaching process was carried out twice at 70 degrees Celsius for 2 hours in each stage. The extracted cellulose was washed and dried using a freeze dryer (Operon FDB-550 model, made in Korea) [24]. The cellulose extraction yield was calculated using equation 4.

$$\text{Cellulose yield (\%)} = 100 \times \left(\frac{C}{Cp}\right) \qquad [4]$$

where C is the amount of extracted cellulose (grams) and CP is the initial amount of corn wood (grams).

2.6. Production of Carboxymethyl Cellulose from Extracted Cellulose

Initially, 100 milliliters of 99 percent isopropyl alcohol was added to 2 grams of dry cellulose, and after stirring, 20 milliliters of 40 percent sodium hydroxide was added. The solution was stirred for 90 minutes. Subsequently, 3 grams of monochloroacetic acid was gradually added to the solution over 30 minutes, and the suspension was stirred for an additional 3 hours at 55 degrees Celsius. Following this, 70 percent methanol was added to the previous contents, and neutralization to pH 7 was performed using 90 percent acetic acid.

The resulting suspension was filtered and washed with 70 percent ethanol through 6 stages and finally with pure methanol. The resulting paste was dried at 60 degrees Celsius and then ground [25].

2.6.1. Physicochemical Properties of Carboxymethyl Cellulose

Moisture content determination was performed through drying using an electric oven (Memmert oven, model UL 40, Schwabach, made in Germany). Ash content was determined using an electric furnace (Ex.1200.2L, Excition Co., made in Iran) at 550 degrees Celsius. The viscosity of 1 percent carboxymethyl cellulose solution was measured using a Brookfield viscometer according to the method of Khullar et al. (2005) [26].

2.6.2. Determination of Degree of Substitution of Carboxymethyl Cellulose

For this purpose, carboxymethyl cellulose ash (0.5 grams at 700 degrees Celsius) was first prepared. The resulting ash was cooled to room temperature in a desiccator. Subsequently, 6 milliliters of deionized boiling water was added to completely dissolve it. The resulting solution was titrated with 0.1 normal sulfuric acid until the solution pH reached 4.4. Finally, the degree of substitution was calculated using equation 5 [25].

$$\text{DS} = \frac{0.162\left(\frac{0.1b}{G}\right)}{1 - 0.08\left(\frac{0.01b}{G}\right)} \qquad [5]$$

where b is the amount of acid required for titration (milliliters) and G is the amount of carboxymethyl cellulose used (grams).

2.6.3. Determination of Carboxymethyl Cellulose Purity

Three grams of sample was precisely weighed and transferred to a 400-milliliter beaker, and 150 milliliters of 80 percent ethanol that had been heated to 60 to 65 degrees Celsius was added to it and placed in a steam bath at 60 to 65 degrees Celsius and stirred with a stirrer for 10 minutes. Subsequently, stirring was stopped and the beaker was placed in the steam bath at rest to allow insoluble materials to settle, and the supernatant was separated. Then 150 milliliters of 80 percent ethanol at 60 to 65 degrees Celsius was added to the beaker and the previous stage was repeated. After material sedimentation, all insoluble materials were transferred to a capped container and 250 milliliters of 80 percent ethanol at 60 to 65 degrees Celsius was added to it and filtered under vacuum and washed with 50 milliliters of 95 percent ethanol. After alcohol evaporation at room temperature, the remaining materials in the container were dried at 105 degrees Celsius for 1 hour. The purity percentage of carboxymethyl cellulose was calculated from equation 6 [27].

$$\text{CMC} = (A\times10000)/(B\ (100-\%C)) \qquad [6]$$

where A is the weight of dried residue, B is the weight of sample used, and C is the moisture percentage of the sample.

2.7. Fourier Transform Infrared Spectroscopy

The Fourier transform spectra of lignin compounds, starch, and saffron corm extract were determined to identify functional groups in the synthesized compounds using a Fourier transform spectrometer (Perkin Elmer, made in USA) in the range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1} [28].

3. Results and Discussion

3.1. General Composition of Saffron Corm and Corn Residues

The physicochemical characteristics of dried saffron corm and corn residues (corncob) are presented in Table 1. As shown in Table 1, total carbohydrates constitute the primary component of both dried saffron corm and corncob. Previous research has examined and reported changes in the content of total soluble sugars and soluble proteins in dried saffron corm during the dormancy period, revealing that total soluble sugar content varies from approximately 6 percent in May to about 13 percent in August, while total protein content ranges from approximately 0.2 percent on a fresh weight basis in May to about 1.2 percent on a fresh weight basis in August [29]. It should be noted that the moisture content of fresh saffron corm was approximately 71.89±0.06 percent.

Table 1 The composition of saffron corm and corn waste

property (grams per 100 grams)	corncob waste (corn wood)	Dried saffron corm
Moisture	7.67 ± 0.12	9.56 ± 0.21
Protein	3.10 ± 0.23	3.29 ± 0.18
Ash	4.93 ± 0.053	1.93 ± 0.06
Lipid	0.06 ± 0.01	3.12 ± 0.03
Fiber	25.70 ± 0.12	1.93 ± 0.01
Total Carbohydrates	58.54 ± 0.51	80.11 ± 0.32

*Data are the average of three replicates and are expressed as mean ± standard deviation.

3.2. Antioxidant Activity of Saffron Corm Extract and Lignin

Table 2 shows the content of phenolic compounds and antioxidant activity of the extract obtained from saffron corm and lignin extracted from corn residues.

Table 2 Phenolic compounds and antioxidant activity in saffron corm and lignin extract.

property	The extract obtained from saffron corm	Lignin extracted from corncob waste
Total phenolic compounds (mgGAE/ 100gDW)	137.91 ± 0.88	156.69 ± 1.59
Radical scavenging activity (DPPH) (%)	29.31 ± 1.04	58.85± 0.69
Ferric reducing antioxidant power (FRAP)(mmol Fe(II)/100g)	5.24 ± 0.88	7.85 ± 0.88
Extraction yield (%)	14.56 ± 2.8	17.32 ± 1.83

*Data are the average of three replicates and are expressed as mean ± standard deviation.

The extraction yield of saffron corm extract and lignin extracted from corn residues in this study was over 14 and 17 percent, respectively.

The successful extraction of biologically active compounds largely depends on the type of solvent used. Water, short-chain alcohols (from methanol to hexanol), halogenated solvents (chloroform, dichloromethane, and carbon tetrachloride), ketones such as acetone and methyl isobutyl ketone, pyridine, 1,4-dioxane, ethyl acetate, diethyl ether, petroleum ether, hexane, and toluene are common solvents employed for extracting various bioactive compounds [30]. The characteristics of a good solvent for extract preparation include low toxicity, ease of evaporation at low temperatures, rapid physiological absorption of the extract, protective function, and minimal interference with extract separation. Factors such as the amount of extractable compounds, extraction speed, diversity of different extracted compounds, variety of extraction-inhibiting compounds, ease of extract application, solvent toxicity in biological assay processes, and potential health hazards of substances present in the extract also influence solvent selection. Furthermore, solvent choice depends on the required extract and the compounds that need to be present in the extract [31]. Studies examining lignin content from corncob and comparing results are very limited. The separation method, extraction technique, and harvest timing affect the lignin content of different corn plant organs. Lignin content has been reported for corn stem (20.45 percent) [32], leaves (16 percent) [33], husk (7.9 percent) [34], grain (20.3 percent) [35], and roots (17.6 percent) [36]. Additionally, different values have been reported for lignin content in corncob cultivated in various countries. The lignin content of corncob grown in Italy, the United States, Malaysia, Turkey, and China has been reported as 4.8, 10.3, 11.3, 15.5, and 19.6 percent, respectively [37,38].

Comparison of these research results with the findings of the present study indicates a high yield of lignin extraction from corn residues and is consistent with the results of other researchers.

The antioxidant capacity of compounds is measured using various methods.

Antioxidants with higher iron-reducing power possess greater capacity to terminate destructive radical chain reactions. The antioxidant activity of saffron corm extract and lignin has been reported in numerous studies. Ain Afshar (2018) reported that saffron corm extract obtained with ethanol solvent had the highest phenolic compounds (85.84 mg per milliliter), iron chelating power (594.74 micromoles per milliliter), and radical scavenging activity (59.42 percent), while ultrasonic processing at 100 percent intensity for 40 minutes extracted the highest level of phenolic compounds (82.23 mg per milliliter) [39]. Lignin is a polymer composed of aromatic subunits with antioxidant activity [40].

3.3. Characteristics of Starch Extracted from Saffron Corm Residues

The chemical composition and extraction yield of starch obtained from saffron corm residues are reported in Table 3.

Table 3 Physicochemical properties of starch extracted from saffroncorm waste

property		* Saffron corm starch
Ash (g/100g)		0.16 ± 0.02
Protein (g/100g)		0.75 ± 0.04
Lipid (g/100g)		0.91 ± 0.03
Amylose (g/100g)		15.78 ± 2.03
Color indices	L*	80.21 ± 2.12
	a*	0.17 ± 0.01
	b*	6.18 ± 0.19
Extraction yield (g/100g)		45.47 ± 2.14

*Data are the average of three replicates and are expressed as mean ± standard deviation.

The ash and crude protein content of the starch sample was 0.16 and 0.75 percent, respectively. The low amounts of ash and protein present in the starch indicate its purity. The amylose content of saffron corm starch was 15.78 percent. Esmaeelian et al. (2022) reported the amylose content of corm as 31.6 percent [41].

The amylose content can vary depending on plant growth conditions and according to plant species or even cultivar [42]. Amylose significantly affects the functional properties of starch. High amylose content influences retrogradation susceptibility and the greater elasticity of starch paste and gel [43]. The L*, a*, and b* values for saffron corm starch powder are shown in Table 4. Consumer preference favors the selection of lighter-colored starch, and plant pigments and polyphenolic compounds have a significant impact on starch quality. The presence of pigments in starch, when transferred to the final product, reduces product quality and acceptability [44]. The L* value has been reported as approximately 89 for bamboo starch [45] and 83 to 90 for acorn starch [46]. The L*, a*, and b* values for starch powder are shown in Table 3. The residual protein content affects the L* value and b* value [47].

3.4. Characteristics of Cellulose and Carboxymethyl Cellulose Obtained from Corn Residues (Corncob)

The extraction yield of cellulose from corncob residues was determined to be 38.43±3.50 percent. The chemical composition of carboxymethyl cellulose prepared from extracted cellulose is reported in Table 4.

Table4 Characteristics of carboxymethyl cellulose from corncob waste cellulose.

property		carboxymethyl cellulose*
Ash (g/100g)		4.75 ± 0.75
Purity grades (%)		73.00 ± 2.42
Degree of substitution (%)		85.01 ± 2.37
Moisture (g/100g)		4.49 ± 0.75
Viscosity of 1% solution (cP)		10.74 ± 0.59
Color indices	L*	83.21 ± 0.852
	a*	0.25 ± 0.29
	b*	759 ± 1.56

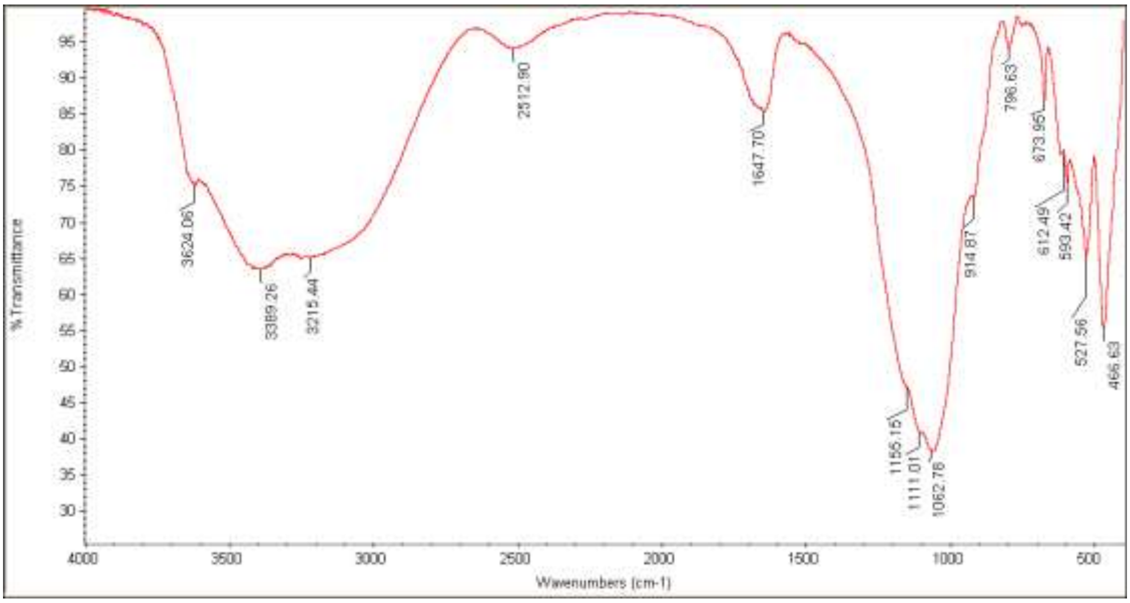
*Mean ± standard deviation

The extraction yield of cellulose from corn residues was approximately 38 percent. Cellulose is the most abundant polymer and the most common organic compound. About 33 percent of total plant mass consists of cellulose as a major component of the cell wall. Cellulose content in cotton, wood, wheat straw, rice straw, sugarcane bagasse, palm leaf, sawdust, and corn residues has been reported as 90, 40 to 50, 28 to 32, 30 to 35, 40 to 45, 20 to 30, 35 to 45, and 40 to 46 percent, respectively [48]. Considering that cellulose content in various tree woods ranges between 45 to 60 percent, it can be stated that the cellulose content in the waste source of corncob is competitive with that found in tree wood. Additionally, different values have been reported for cellulose content in corncob cultivated in various countries. The cellulose content in corncob cultivated in Italy, Turkey, the United States, and Malaysia has been reported as 36.4, 52, 45.2, and 45.9 percent, respectively [37,49]. Additionally, the cellulose content of corncob has been reported to vary based on its color. The cellulose content in white and yellow corncob from Nigeria has been reported as 33.6 and 33.1 percent, respectively [38,50].

The degree of substitution, purity level, and viscosity of 1 percent carboxymethyl cellulose solution are shown in Table 4. The degree of substitution is a factor that influences the solubility of carboxymethyl cellulose, and this factor is directly related to changes in sodium hydroxide concentration during the alkali cellulose formation stage. Due to the higher molecular weight of primary hydroxyl groups in the cellulose structure, weight increase occurs during the carboxymethylation reaction. Therefore, as the degree of substitution increases, the production yield of carboxymethyl cellulose also increases. The viscosity and degree of purity of carboxymethyl cellulose products are among the most important factors affecting their range of applications. The degree of crystallinity and degree of polymerization of cellulose affect the amount of carboxymethyl cellulose [51].

3.5. Fourier Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy is widely used in studies of various compounds because it provides a simple method for obtaining information about chemical changes that may occur during chemical treatment. To confirm the substitution of carboxymethyl groups on cellulose and the production of carboxymethyl cellulose, Fourier transform infrared spectroscopy was used and compared with the spectroscopy of pure cellulose (Figure 2).



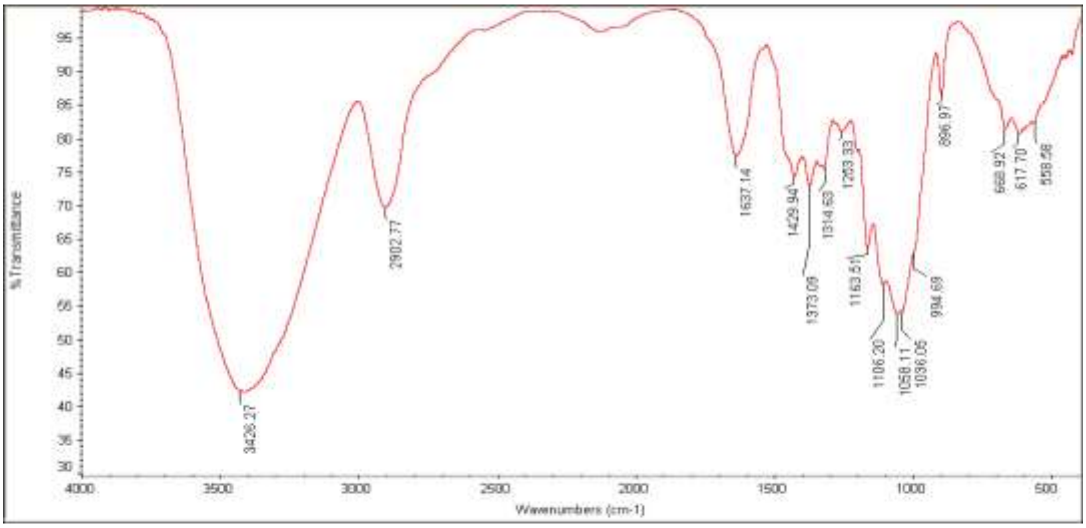


Fig 2 Fourier-transform infrared spectra of A: cellulose B: carboxymethyl cellulose from corncob residues

Examination of the infrared spectrum of cellulose extracted from corn residues shows that the most important peaks were observed at wavenumbers 3389, 3215, 2512, 1647, 1155-1111, and 1062 cm⁻¹. The peak observed at 3389 to 3215 cm⁻¹ corresponds to O-H stretching of water absorbed on cellulose as well as intramolecular and intermolecular hydrogen bonds of cellulose. The band observed at wavenumber 2512 cm⁻¹ indicates asymmetric C-H stretching present in cellulose and hemicellulose. The C=O stretching of the ether group present in hemicellulose can be observed at wavenumber 1647 cm⁻¹. The range from 1155 to 1111 cm⁻¹ may correspond to angular deformation of C-O bonds in primary alcohols. Additionally, the absorption band observed at wavenumber 1062 cm⁻¹ shows the C-O-C bonds characteristic of cellulose pyranose ring vibration. The results of the present study were consistent with the findings of Reis et al. (2020), who also reported the presence of various peaks at wavenumbers 3335, 2910, 1730, 1375, 1160, and 1020 cm⁻¹ in the spectroscopy of cellulose microfibrils extracted from coffee [52]. Examination of the infrared spectrum of carboxymethyl cellulose reveals a broader band in the wavenumber range of 3426 cm⁻¹ and the appearance of peaks in the wavenumber range of 1637 to 1163 cm⁻¹ compared to the infrared spectrum of cellulose. The broader band in the 3426 cm⁻¹ range indicates an increase in hydroxyl groups in carboxymethyl cellulose compared to cellulose. The band corresponding to the 2902 cm⁻¹ region also shows the presence of methyl groups, which were also evident in cellulose within the same ranges. The peaks observed at wavenumbers 1637 to 1253 cm⁻¹ are due to the presence of carboxyl groups and their salts, which confirms the substitution of carboxymethyl groups in the cellulose structure. The results of the present study were consistent with the findings of Rahman et al. (2022) [53]. Li et al. (2019) also reported strong absorption bands in carboxymethyl cellulose derived from wheat straw at wavenumbers 1605 and 1420 cm⁻¹ [54].

Lignin is an amorphous polymer composed of phenylpropane units, and its precursors are three alcohols: p-coumaryl, coniferyl, and sinapyl, whose main function is to strengthen plant structure, provide impermeability, and confer resistance against microbial attacks and oxidative stresses [55].

Figure 3 shows the infrared spectrum of lignin. The broad absorption band at wavenumber 3413 cm⁻¹ is attributed to O-H stretching vibration, while the band observed at 2934 cm⁻¹ is assigned to symmetric C-H vibration of the methylene group (-CH2). The peaks at 1709 and 1631 cm⁻¹ correspond to stretching vibrations of unconjugated carbonyl and conjugated carbonate, representing carboxylic acid and ketone groups, respectively. The peaks at 1603, 1513, and 1423 cm⁻¹ indicate aromatic ring vibrations. The peak at 1462 cm⁻¹ is attributed to methoxyl C-H bending and C-C stretching in the aromatic skeleton. The absorption peaks at 1167, 1128, and 832 cm⁻¹ represent p-hydroxyphenylpropane (H), syringyl (S), and guaiacyl (G) units, respectively, in HGS-lignin. The bands at 1329 and 1260 cm⁻¹ were attributed to aromatic C-O stretching of S units and/or G units condensed at G5 (Figure 3) [56].

The results of infrared spectrum analysis of cellulose, carboxymethyl cellulose, and lignin extracted from the wood of two tree species (Calycophyllum spruceanum and Guazuma crinita Lam.) were also consistent with the findings of this study [57]. These researchers attributed the minor differences in the wavenumber range related to various bands and groups to minor differences present in the structure of these compounds as well as the method and manner of extraction.

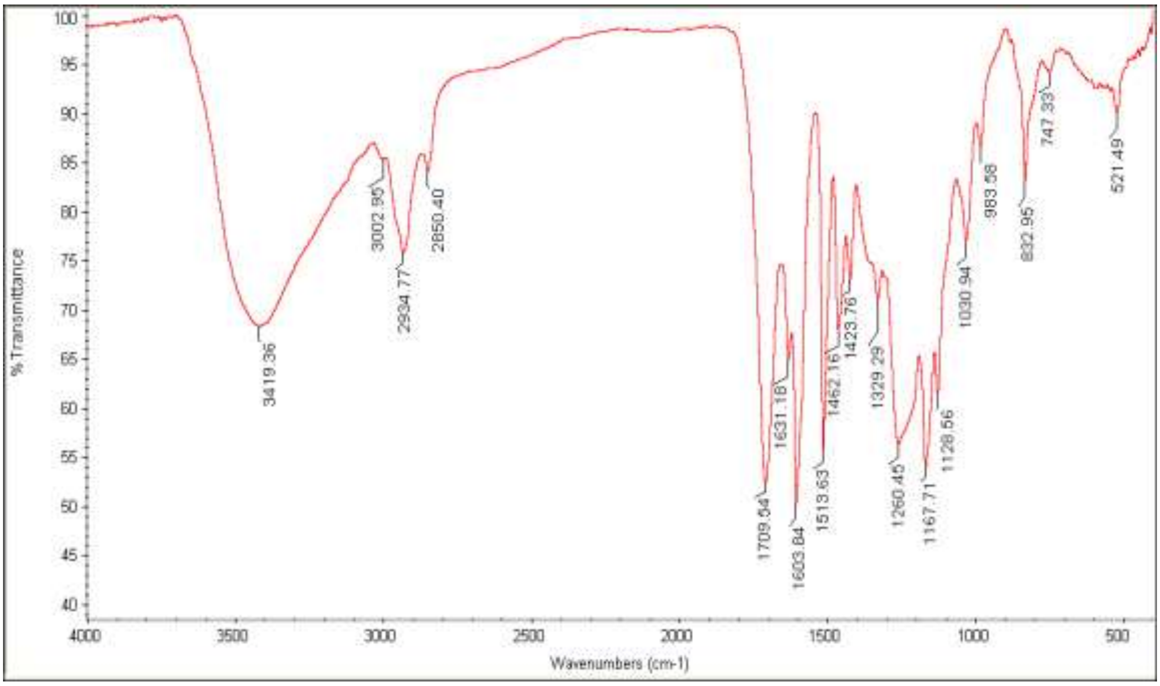


Fig 3 Fourier-transform infrared spectra of Lignin extracted from corncob waste

In order to compare the molecular structure of starch obtained from saffron corm under study, infrared spectra of the samples were prepared, which are presented in Figure 4. The infrared spectrum included three main regions, although peaks were also observed outside these regions. The first main region (3600 to 3000 cm^{-1}), which is the main large peak on the left side of the graph, was related to symmetric and asymmetric stretching of the hydroxyl functional group, and due to the abundance of this functional group in the constituent molecules of starch as well as the presence of bound water molecules, extensive absorption was observed in this region [58]. The second main region (3000 to 2800 cm^{-1}) was related to asymmetric CH stretching, and due to the extensive presence of glucose molecules in the starch structure, this peak was observed. The peak in the 1700 to 1600 cm^{-1} region was due to bending of water molecules absorbed in the amorphous region of starch (Figure 4). Increasing the ratio of the crystalline region causes a decrease in peak intensity in this region [59]. The third region (800 to 1500 cm^{-1}), known as the fingerprint region, consists of several sections. The section from 1500 to 1300 cm^{-1} included peaks related to CH bending, CH2 bending, and C=O stretching [58]. The range from 1200 to 1000 cm^{-1} is known as the carbohydrate peak and is affected by glucose vibrations as the structural unit of starch, and its chain arrangement, such as double helix formation and crystalline regions, changes the absorption intensity in this region. The three main peaks in this region were due to stretching vibrations of the C-O-C bond of the glycosidic bond and H-O-C adjacent to this bond. Additionally, the peak observed at wavenumber 855 cm^{-1} corresponds to alpha anomeric bonds, which are the main bonds in the formation of amylose and amylopectin polymers [60].

Fourier transform infrared spectroscopy can be a suitable method for detecting the degree of order in starch through studying disorder-to-order transitions in starch retrogradation reactions. It has been reported that the peak at wavenumber 1047 cm^{-1} is sensitive to order or crystalline starch, while the absorption band at 1022 cm^{-1} is related to amorphous structure in starch. Therefore, the intensity ratio of wavenumbers 1047 to 1022 cm^{-1} can indicate the ratio of crystalline (ordered) starch to amorphous starch [61].

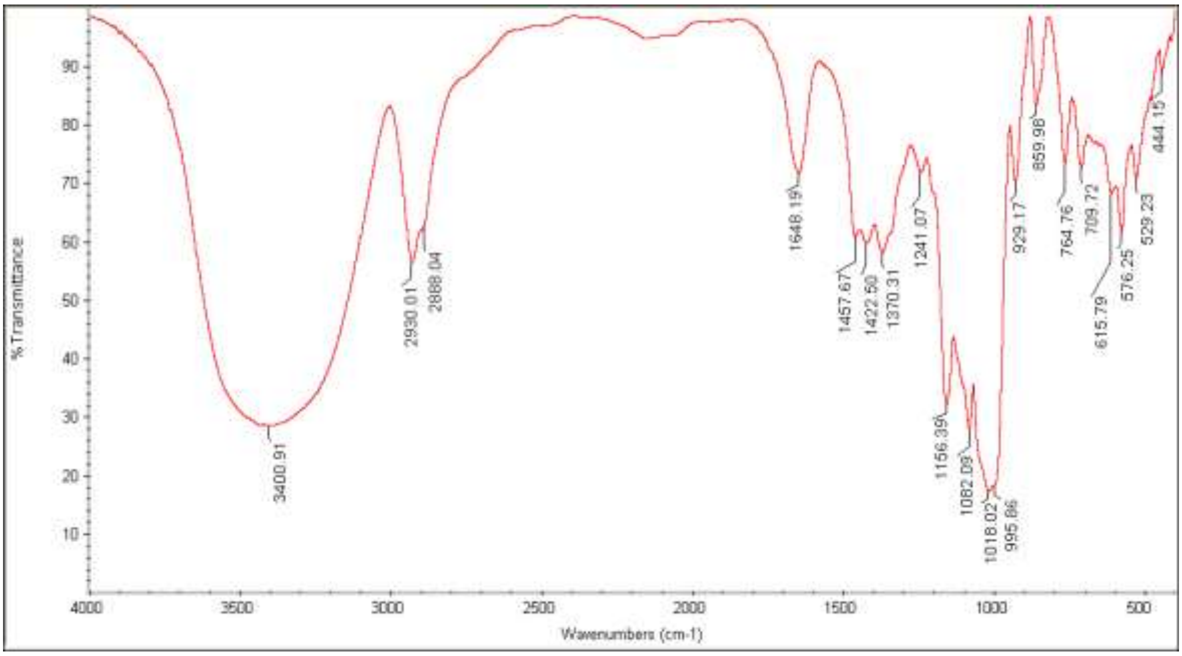


Fig 4 Fourier-transform infrared spectra of starch extracted from saffron corm

3.6. Investigation of Antimicrobial Effect of Saffron Corm Extract and Lignin

3.6.1. Results of Microdilution Method

The results of minimum inhibitory concentration and minimum fungicidal concentration of saffron corm extract and lignin on Aspergillus flavus and Aspergillus parasiticus molds are shown in Table 5.

As observed in Table 5, both active compounds possessed antifungal activity. The antifungal effect of saffron corm extract and lignin is likely due to the phenolic compounds present in them.

Phenolic compounds play an important role in preventing the growth of bacteria and fungi, and the degree of effectiveness of these compounds varies depending on the type of phenolic compounds, concentration of phenolic compounds, extraction method, solvent used for extraction, and other factors [62]. The minimum inhibitory concentration and minimum fungicidal concentration of lignin were lower than those of saffron corm extract. Given the higher phenolic compound content of lignin compared to saffron corm extract, this result was not unexpected.

Fig 5 Antimicrobial power based on minimal inhibitory concentration and minimal fungicidal concentration (mg/l)

Type of antimicrobial active compound		Type of microorganism	
		Aspergillus flavus	*Aspergillus parastiticus*
Saffron corm extract	minimal inhibitory concentration	0.25	0.03125
	Minimal Fungicidal Concentration	>0.5	> 0.5
Lignin	minimal inhibitory concentration	0.00019	0.00039
	Minimal Fungicidal Concentration	> 0.05	> 0.05

3.6.2. Results of Disk Diffusion Method

The antifungal effect of saffron corm extract and lignin derived from corncob (concentrations of 0.5 and 0.05 mg per liter) on Aspergillus parasiticus mold using the disk diffusion method on potato dextrose agar culture medium is shown in Figure 5.

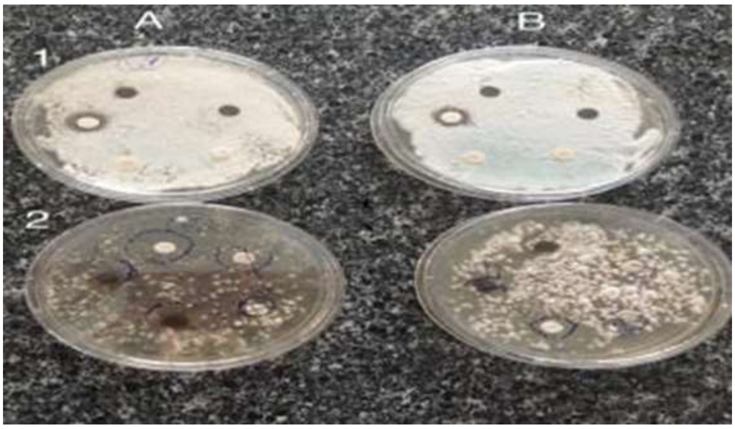


Fig 5 The inhibition halo of saffron corm extract and lignin (Concentrations of 0.5 and 0.05 mg/l) on *Aspergillus parasiticus* (1 and 2 respectively the concentrations of 10^6 and 10^3 colony forming units per gram of *Aspergillus parasiticus* and A and B respectively lignin and saffron corm extract)

Table 6 shows the effect of saffron corm extract and lignin compared to ketoconazole fungicide and solvents used to dissolve the active compounds on the mean diameter of growth inhibition zones of Aspergillus flavus and Aspergillus parasiticus molds.

Fig 6 The effect of saffroncorm extract and lignin in comparison with Ketoconazole on the average diameter of theinhibition halo (mm)

Material type	Type of microorganism	
	Aspergillus flavus	spergillus parastiticus
Saffron corm extract	13.75 ± 2.05^{b}	10.00 ± 1.41^{b}
Lignin	16.00 ± 1.63^{b}	9.50 ± 0.57^{b}
Ketoconazole (200 ppm)	20.50 ± 0.5^{a}	12.00 ± 1.00^{a}
Dimethyl sulfoxide	-	-
water	-	-

Data are the average of three replicates and are expressed as mean ± standard deviation.
Non-common letters in each column indicate a significant difference at the 5% level (Duncan's test, $P<0.05$).

The results indicated antifungal effects of saffron corm extract and lignin. The solvents dimethyl sulfoxide and water showed no antifungal effects. Lignin possessed greater antifungal properties compared to saffron corm extract and showed a larger growth inhibition zone diameter. Additionally, both active compounds showed greater antifungal effects against Aspergillus parasiticus. The antimicrobial effect of plant extracts and lignin is attributed to the presence of polyphenols [63]. The effect of plant extracts as antimicrobial agents depends on the type of polyphenols such as phenolic acids, flavonoids, and tannins. Additionally, the antimicrobial activity of phenolic compounds is influenced by the position and number of hydroxyl groups, as these groups react with microbial cell walls and cause disruption in the cell wall and leakage of cellular compounds [64]. Rubio-Moraga et al. (2013) reported the antifungal effect of extracts from inner and outer portions of saffron corm against Aspergillus niger, Bipolaris spicifera, Fusarium oxysporum, Penicillium restrictum, and Rhizopus nigricans. Additionally, antibacterial effects of lignin against gram-positive bacteria (Bacillus and Klebsiella) and gram-negative bacteria (Escherichia coli and Staphylococcus aureus) have been reported [3].

4. General Conclusion

Overall, the results showed that the phenolic compound content and free radical scavenging activity of saffron corm extract and lignin were 137.91 (mg per 100 g dry weight of corm) and 29.31 percent, and 156.69 (mg per 100 g dry weight of corncob) and 58.85 percent, with extraction yields of 14.56 and 17.32 percent, respectively. The mean diameter of growth inhibition zones of Aspergillus flavus and Aspergillus parasiticus molds due to saffron corm extract and lignin were 13.75 and 10 mm, and 16 and 9.5 mm, respectively. The starch obtained from saffron corm also possessed desirable characteristics, as the protein and ash content of saffron corm starch were low at 0.16 and 0.75 percent, respectively (close to potato starch and slightly lower than rice starch). The low amounts of ash and protein present in the starch indicate its purity. The amylose content of saffron corm starch was 15.78 percent. The extraction yield of cellulose from corn residues was approximately 38 percent, and the carboxymethyl cellulose prepared from it had a degree of substitution of 0.85, which indicated favorable production yield. To confirm the substitution of carboxymethyl groups on cellulose and the production of carboxymethyl cellulose, Fourier transform infrared spectroscopy was used and compared with the spectroscopy of pure cellulose. The results confirmed the substitution of carboxymethyl groups in the cellulose structure (observation of peaks at wavenumbers 1637 to 1253 cm^{-1}, indicating the presence of carboxyl groups and their salts). Additionally, Fourier transform infrared spectroscopy of starch and lignin also confirmed the ordered and coherent structure of these compounds. Based on the results of this research, it can be stated that residues and wastes from corn and saffron corm are valuable and potential sources for producing high value-added products, for which significant amounts of foreign currency are annually spent to import them into the country, which emphasizes and highlights the necessity of attention and investment in this field more than ever.

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بررسی ویژگی‌های لیگنین، کربوکسی‌متیل سلولز و نشاسته استخراج شده از زائدات ذرت و ضایعات پیاز زعفران

راضیه نیازمند[۱*]؛ پروین شرایعی[۲*]؛ وحیده شریف[۳]؛ محبوبه سرابی جماب[٤]؛ سودابه عین افشار[۲]؛ الهام آذرپژوه[۲]؛ فرزانه شریف[۳]

۱-گروه شیمی مواد غذایی، مؤسسه پژوهشی علوم و صنایع غذایی، مشهد، ایران.

۲-بخش تحقیقات فنی و مهندسی کشاورزی، مرکز تحقیقات و آموزش کشاورزی و منابع طبیعی استان خراسان رضوی سازمان تحقیقات، آموزش و ترویج کشاورزی،مشهد، ایران.

۳-پژوهشکده زعفران دانشگاه تربت حیدریه، تربت حیدریه، ایران.

٤-گروه بیوتکنولوژی مواد غذایی، موسسه پژوهشی علوم و صنایع غذایی، مشهد، ایران

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[*] مسئول مکاتبات:

r.niazmand@rifst.ac.ir

parvin_sharayei@yahoo.com

چکیده

این پژوهش با هدف تولید محصولات با ارزش افزوده بالا شامل نشاسته و کربوکسی‌متیل‌سلولز به ترتیب از بنه‌ی زعفران و ضایعات ذرت و همچنین تولید عصاره‌های فعال از بنه‌ی زعفران و لیگنین از ضایعات ذرت انجام شد. در این مطالعه بازده استخراج، ظرفیت آنتی‌اکسیدانی و خاصیت ضد قارچی عصاره‌های حاصل از بنه‌ی زعفران و لیگنین تهیه شده از ضایعات ذرت و همچنین ویژگی‌های فیزیکوشیمیایی نشاسته‌ی حاصل از بنه‌ی زعفران و کربوکسیل متیل سلولز تهیه شده از چوب ذرت بررسی شدند. بازده استخراج عصاره‌ی بنه‌ی زعفران و لیگنین استخراج‌شده از ضایعات ذرت به ترتیب بیشتر از ۱٤ و ۱۷ گرم در صد گرم و میزان ترکیبات فنلی و فعالیت گیرندگی رادیکال آزاد عصاره بنه زعفران و لیگنین به ترتیب ۱۳۷/۹۱ (میلی‌گرم معادل گالیک اسید در ۱۰۰ گرم وزن خشک بنه) و ۲۹/۳۱ درصد و ۱۵٦/٦۹ (میلی‌گرم معادل گالیک اسید در ۱۰۰ گرم وزن خشک چوب ذرت) و ۵۸/۸۵ درصد بود. حداقل غلظت ممانعت‌کنندگی و حداقل غلظت قارچ‌کشی لیگنین به دلیل بالاتر بودن میزان ترکیبات فنلی آن، پایین‌تر از عصاره‌ی بنه‌ی زعفران بود. میانگین قطر هاله عدم رشد کپک‌های *آسپرژیلوس فلاووس* و *آسپرژیلوس پارازیتیکوس* در اثر عصاره بنه زعفران و لیگنین به ترتیب ۱۳/۷۵ و ۱۰ میلی‌متر و ۱٦ و ۹/۵ میلی‌متر بود. محتوای خاکستر و پروتئین نشاسته‌ی حاصل از پیاز زعفران به ترتیب ۰/۱٦ و ۰/۷۵ گرم در صد گرم بود. بازده استخراج سلولز از ضایعات چوب ذرت ۳۸/٤۳ گرم در صد گرم و میزان خلوص و درجه‌ی استخلاف کربوکسی‌متیل‌سلولز تهیه شده از آن به ترتیب ۷۳ و ۸۵ گرم در صد گرم بود. نتایج حاصل از پرتوسنجی تبدیل فوریه زیرقرمز، تشکیل کربوکسی‌متیل‌سلولز از سلولز استخراج شده از چوب ذرت را تایید کرد. با توجه به نتایج پژوهش حاضر می توان گفت که ضایعات و زائدات مواد غذایی به عنوان منابع پایدار و کم هزینه برای تولید محصولات ارزشمند مانند سلولز و نشاسته با کاربردهای صنعتی قابل استفاده هستند.