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Original research

Application of Orange Pomace as a Low-Cost Biosorbent in Removing Heavy Metals from Brown *Oryza Sativa* Rice

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A B S T R A C T

Rice, beyond being a staple carbohydrate source, is a treasure trove of valuable nutrients and bioactive compounds with significant health benefits. It contains essential nutrients like dietary fiber, minerals, protein, and B vitamins, along with potent phytochemicals such as tocols (tocopherols and tocotrienols), oryzanol, phenolic acids, and flavonoids. These compounds contribute to rice's antioxidant, anti-inflammatory, anticancer, and hypoglycemic properties, making it a potential functional food and nutraceutical. Brown rice can contain higher levels of certain heavy metals compared to white rice. The reason brown rice tends to have higher heavy metal levels than white rice is that the bran layer, which is removed during processing to make white rice, is where these metals accumulate in the rice grain. Biosorption is indeed a method used to remove heavy metals from consumable rice, where the metals bind to the surface of insoluble compounds (biosorbents) within the rice. These biosorbents can be derived from various sources, like agricultural waste, microorganisms, or algae. The process involves heavy metals being adsorbed onto the pores and surfaces of these materials, effectively removing them from the rice. The study investigated the potential of orange pomace, a byproduct of orange processing, to remove lead and cadmium from Tarom-Hashemi brown rice. The study findings indicate that orange pomace, particularly due to its pectin content, exhibits a significant capacity to adsorb lead (Pb) and cadmium (Cd) from aqueous solutions and potentially detoxify Tarom brown rice. This achievement is supported by the fact that the adsorption of these heavy metals is statistically significant ($p < 0.003$), and the process is likely driven by the pectin's chemical properties. Using readily available agricultural and food industry waste, specifically pomaces, is a promising and cost-effective method for detoxifying high-consumption rice in Iran.

Keywords: Brown Rice; Detoxification; Agricultural waste; Orange Pomace; Nutraceutical

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1. Introduction

Rice is a crucial staple food globally, especially in Asia, and it holds significant importance in Iran, ranking as the second most important crop and a fundamental part of daily sustenance in the country. Its role extends beyond mere sustenance, contributing to food security and cultural identity. Rice contains various bioactive compounds, including phenolic acids, flavonoids, and γ-oryzanol, which contribute to its potential health benefits. These compounds, particularly in pigmented rice varieties, exhibit antioxidant, anti-inflammatory, and anticancer properties (Zubair et al., 2023).

Rice is a popular source of carbohydrates, also contains a good amount of dietary fiber, minerals (Ca, Zn, Se, P, K, Mg, Fe, and Mn)(Ziarati& Azizi, 2014), protein, and vitamin B along with several other medicinally important bioactive compounds such as: tocols (α-tocopherols and α-tocotrienols) (ßsitosterol) phenolic acids, flavonoids (apigenin). Its components do indeed possess strong antioxidant activity, which helps to neutralize harmful free radicals and mitigate oxidative stress. This antioxidant capacity is attributed to various bioactive compounds present in rice, including

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phenolic acids, flavonoids, anthocyanins, and others. These compounds can effectively scavenge free radicals and reduce the production of reactive oxygen species (ROS), thereby protecting cells and tissues from oxidative damage (Goufo & Trindade 2014; Kang et al., 2022). Brown rice is a whole grain that contains the bran layers and embryo, which are rich in various nutritional and biofunctional components. These include dietary fibers, γ-oryzanol, vitamins, and minerals. These components are primarily located in the bran and germ of the rice grain (Cho & Lim, 2016; Ravichanthiran et al., 2018; Ziarati et al., 2022).

Brown rice is more nutritious than white rice, but it can also contain higher levels of heavy metals, particularly arsenic, due to its outer bran layer. While brown rice offers more fiber, vitamins, and minerals, it also absorbs more contaminants from the soil and water during growth, including arsenic (Gray et al., 2016; Kumarathilaka et al., 2019). Recent studies have indeed indicated higher levels of heavy metals, particularly arsenic, in brown rice (Oryza sativa) compared to white rice. This is a concern because heavy metals like arsenic, cadmium, and lead can pose health risks, especially with long-term exposurerecent studies have indeed indicated higher levels of heavy metals, particularly arsenic and cadmium in brown rice (Oryza sativa) compared to white rice. This is a concern because heavy metals like arsenic, cadmium, and lead can pose health risks, especially with long-term exposure (Mridha et al.,2022). Recent studies have indeed shown elevated levels of lead (Pb) and cadmium (Cd) in brown rice (Oryza sativa). These findings raise concerns about potential health risks associated with consuming brown rice, as both lead and cadmium are toxic heavy metals (IARC . 2012.; Gorgani & Ziarati , 2020).

Although current evidence supports the fact that the consumption of brown rice is beneficial for health, some studies have revealed that brown rice can be a source of concern for Arsenic, lead and cadmium contamination, and these heavy metals can pose potential health risks. While brown rice is often preferred for its nutritional value, including higher fiber content compared to white rice, its outer layers can accumulate more heavy metals, including lead and cadmium, from the soil during growth (Saadatzadeh et al., 2023; Toledo et al., 2022).

Heavy metal contamination of soil stems from both natural and human-induced sources. Natural sources include weathering of rocks, volcanic eruptions, and forest fires. Anthropogenic sources, which are far more significant, include industrial activities (mining, smelting, manufacturing), agricultural practices (use of fertilizers, pesticides, and sewage sludge), and urban activities (vehicle emissions, landfill leachate) (Alengebawy et al., 2021). There is a growing concern about heavy metal contamination in rice in Asia as a staple food, and increased screening of rice products is recommended to ensure safety and minimize health risks. Studies highlight the concern that rice can absorb heavy metals like arsenic, cadmium, and lead from soil and water, which can lead to health problems, especially with long-term exposure. These metals can accumulate in the rice grains and enter the food chain, posing risks to human health. Specific investigations on the detoxification of rice are severely suggested as rice can accumulate heavy metals like arsenic, cadmium, and lead from soil and water. These metals can pose health hazards, especially with chronic exposure (Rezaei et al., 2021; Proshad et al., 2020). Chronic, low-level exposure to heavy metals like arsenic, cadmium, and lead can indeed lead to various health issues, including neurological disorders and hypertension, among other problems (Kothapalli, 2021; AlMulla et al., 2022).

Several studies indicate that low-cost fruit and agricultural waste can be utilized effectively to detoxify heavy metals from contaminated soils, rice, and other food sources. Agricultural waste materials like rice husk, orange peels, and pistachio bio-waste have shown promise as bio-adsorbents for removing heavy metals such as lead, cadmium, and arsenic from rice and wastewater(Gholizadeh, E. & Ziarati, 2016; Motaghi & Ziarati,2016; Lahiji et al., 2016).

Orange pomace (OP), a byproduct of orange juice production, is a versatile material with potential applications in various fields. It is rich in fiber, bioactive compounds, and other valuable components, making it a promising resource for industries like food, pharmaceuticals, and agriculture. Orange pomace typically includes peel, seeds, and pulp. This pomace is rich in valuable bioactive components like pectin and cellulose. Additionally, it contains other carbohydrates like hemicellulose and simple sugars. Many projects and research recently Citrus fruit processing waste (CFPW) is a valuable source for sustainable mitigation due to its potential to be a bioresource for biopolymers and bioactive compounds. These wastes can be processed to extract pectin, cellulose, hemicellulose, lignin, and bioactive compounds with antioxidant and antimicrobial properties. This utilization aligns with the principles of a circular economy and zero waste, contributing to environmental sustainability (Dubey, et al., 2023; Castagna, et al., 2025; Kato-Noguchi, & Kato 2025). Citrus waste can be utilized in many ways beyond organic fertilizer, including as a feedstock for energy production, a source of bioactive compounds, and a bio-absorbent. Other non-food applications include use as a biofertilizer, in biodiesel production, and for biogas and bioethanol generation (Abbasi, et al., 2015).

Feeding citrus waste to animals is a common application, particularly in Iran, due to its nutritional content. Citrus waste, including peels and pulp, can be a valuable feed source for various livestock (Idamokoro and Hosu, 2022).

Citrus fruits contain not only essential nutrients but also various non-nutrient, biologically active compounds, such as flavonoids, carotenoids, and other phytochemicals, that have potential medicinal benefits for animals. These compounds contribute to the overall health-promoting properties of citrus fruits, including antioxidant, anti-inflammatory, and anti-cancer effects (Benavente-Garcia and Castillo, 2008; Idamokoro and Hosu, 2022).

Fruit peels, particularly orange peels, can act as effective adsorbents for pollutants due to their abundance of active sites, primarily stemming from surface functional groups like hydroxyl and carboxyl groups, according to research from ScienceDirect and other recognized scientific sites. These groups facilitate the binding of pollutants through various mechanisms, including electrostatic interactions, hydrogen bonding, and complexation, according to a study published on ScienceDirect. Research by Yirga in 2022 explored the use of modified orange peel as a low-cost biosorbent to remove heavy metals from wastewater. The study found that orange peel powder effectively removes copper(II) and cadmium(II) ions from contaminated water, with removal rates of 96.9% and 98.1% respectively, under optimized conditions, according to the research paper (Yirga et al., 2022).

The current research focused on using orange pomace as a low-cost adsorbent to mitigate heavy metal contamination in rice, a staple food. The study aimed to determine how effectively orange pomace could bind to these toxic metals, thus reducing their presence in the rice.

Material and Methods

Rice Sampling Method

Hashemi Tarom rice is primarily cultivated and harvested in the Gilan and Mazandaran provinces of northern Iran. These regions are known for their fertile soil and suitable climate for rice cultivation, particularly for these aromatic and flavorful varieties.

40 samples of Iranian Hashemi-Tarom (Oryza sativa L.) brown rice were purchased randomly from 10 rice paddies in 2020, from Astaneh Ashrafieh, Rasht and Someh Sara in Guillan and Babol, Sari, and Amol rice paddies from Mazandaran provinces in the north of Iran. The Field trials conducted relying on traditional techniques, particularly for land preparation and weed control..

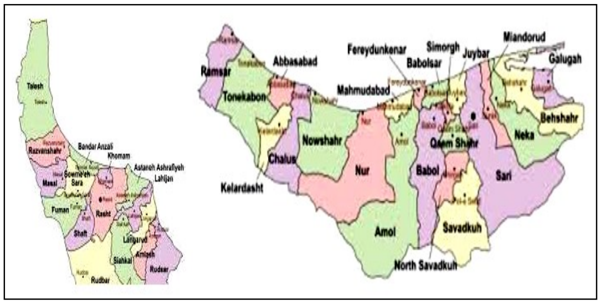

Figure 1- The Overview of the study area: rice-planting areas in Gilan and Mazandaran Provinces

The research involved analyzing characteristics like moisture content, dimensions, weight, and potentially milling properties. The specific procedures used for sample collection, preparation, and analysis, potentially including cooking methods and testing for heavy metals just like previous studies by authors (Razafsha& Ziarati 2016; Gholizadeh, E. & Ziarati, 2016 ; Motaghi & Ziarati,2016; Lahiji et al., 2016;Gorgani & Ziarati , 2020). Five portions of each Oryza sativa rice samples from each farm before milling (whitening), in preliminary round of laboratory analyzing were mixed. All experiments were conducted with 5 replications.

After the initial analysis of cadmium, lead, and nickel contents in raw brown samples from all farms in the two provinces, the next step involved analyzing a composite sample created by mixing all the individual raw samples together. These composite samples were then subjected to the same analytical procedures to determine the heavy metal concentrations, allowing for an overall assessment of contamination levels across all the collected samples.

Orange Pomace Sampling Method

Citrus sinensis, or sweet orange, is a hybrid citrus fruit, commonly used in the production of orange juice and other food products, including functional foods. Citrus sinensis includes varieties like Valencia orange, blood orange, and navel orange. Orange juice is a widely consumed beverage globally. In Tehran, Iran, oranges, including those of the Citrus sinensis species, are popular for juicing and other food applications (Rampersaud and Valim , 2017 ; Zacarías-Garcia et al., 2023;. Seyedi , et al., 2022). The orange processing waste was acquired from Takdaneh Industry Co. in Marand, Iran, in winter 2020 for using in this specified study. The waste likely originates from their orange juice production process. The research focuses on valorizing orange processing byproducts, possibly for applications in the detoxification of heavy metals from brown rice.

Figure 2- The dried powder of Orange Pomace.

The orange pomace (OP) is cut into small pieces by small, clean cutter and naturally dried in sunlight (figure 2). Dried pomace was ground using a clean electric mixer, sieved through (Retsch GmbH & CoKG, Germany) mesh size (250 µm) to retain fine particles. To ensure of main process for preparing waste material for adsorption experiments, focusing on preventing enzymatic browning, the OP were then dipped in a 1% (w/v) citric acid solution for 10 min, drained and dried in an oven at 80°C for 24 hours, and homogenized in a blender to utilize in adsorption experiments. The study aim was to determine the optimal dose of orange pomace powder for detoxifying heavy metals from brown rice, by the purpose: Find the best efficacy as a biosorbent for heavy metal detoxification ; Doses of orange pomace powder: 1, 2, 3, 5, and 10 (W/W).

Cooking methods

a) The rice cooking process is settled by using both the Kateh method, which is an Iranian style of cooking rice with water and fat, and the draining method, a more conventional technique where rice is boiled and then drained. In Kateh, rice was cooked with water and fat until the water was absorbed. In the draining method, rice was boiled until partially cooked, then drained.

b) The rice, after being soaked by or without bio-adsorbent, was first drained, and then was added to boiling water and cooked until partially tender. The cooking process involved boiling the rice in 400ml of water over medium heat for 8-10 minutes. Following this, the water was drained, and the rice was moved to a clean pot. The rice was cooked in a pot with a tight-fitting lid. Cooking lasted for 30-35 minutes, until all the water was absorbed and the rice was fully cooked. No additional ingredients were added during the cooking process. After cooking, the rice was cooled to room temperature and then stored at 4°C for later analysis.

Experiment

Lead and Cadmium concentrations of Raw, Rinsed, soaked by water/salt, soaked by OP(orange pomace powder in figure 1) in different contact time/ NaCl, cooked in both states of treated and untreated by OP association by two different cooking methods were determined by the wet digestion method. The described procedure is a wet digestion method used for preparing rice samples for elemental analysis. It involves using a mixture of concentrated nitric acid (25 mL) and hydrochloric acid (8 mL) with a 10-gram rice sample, and gradually heating the mixture on a hot plate until complete digestion was achieved. This process ensures that all organic matter is broken down, leaving behind a clear solution ready for analysis. The gradual heat increase is crucial to prevent violent reactions and ensure complete digestion (Jafari Moghadam et al., 2016; Lahiji et al., 2016; Ziarati et al., 2022). The determination of nickel, lead, and cadmium content, including any residual amounts, was performed using a flame atomic absorption spectrophotometer (FAAS). The process involved standardized international protocols for sample preparation and analysis, utilizing a wet digestion method and adhering to the annual book of ASTM standards and AOAC guidelines (AOAC, 2000).

All digested sample flasks were first heated slowly and then vigorously till a white residue is obtained. The laboratory procedure continued where a solid residue was dissolved in a solution of 0.1 N nitric acid (HNO3) and then diluted to a final volume of 10 milliliters using a volumetric flask, due to prepare solutions for analysis by atomic absorption spectroscopy.

All glassware and plastic containers used were washed with liquid soap, rinsed with water, soaked in 10% volume/volume nitric acid for 24 hours, cleaned thoroughly with distilled water, and dried in such a manner to ensure that any contamination does not occur. Blanks and samples were also processed and analyzed simultaneously. All the chemicals used were of Analytical Grade (AR). For heavy metal analyses, 5 grams of each rice sample in different states and stages of cooking method were weighed on an electronic balance (Shimadzu LIBROR AEX 200G). The samples were analyzed according to standardized international protocols by wet digestion method, using HNO3 and HCl, analyzed by a Flame Emission Spectrophotometer Model AA-6200 (Shimadzu, Japan) using an air-acetylene flame for heavy metals in Research Analytical Laboratory in Pharmaceutical Sciences Branch, Islamic Azad University, using six standard solutions for each metal. All necessary precautions were taken to avoid any possible contamination of the sample as per the AOAC guidelines (Ziarati, et al., 2020; AOAC, 2020).

Bio-removal of Cadmium & Lead from Rice Samples

According to the results of all experiments applied above, the current investigation was designed to examine the capacity of orange pomace for the bio-removal of cadmium and lead ions from contaminated rice samples after the determination of these metals in such rice samples in different states. In this experiment, 0.100 g powder of dried orange pomace in the form of powder dried pieces, was placed into a plastic tank containing 50 g of rinsing rice samples (5 times by distilled water) and 250 ml of deionized water left under laboratory conditions at pH = 6.3 and 250 C for almost 1 hour. In order to establish a baseline for comparison and assess the effectiveness of a bio-adsorbent, a portion of the samples was intentionally left untreated with the bio-adsorbent. This untreated group serves as a blank and allowing researchers to distinguish between the effects of the bio-adsorbent and any naturally occurring background adsorption or other factors. By comparing the results from the treated and untreated samples, the true impact of the bio-adsorbent can be evaluated (Jafari-Moghadam et al., 2015, and 2016; Razafsha and Ziarati 2016). .

Bio-sorbed metal concentration (mg/kg dry weight) and bio-sorption capacity (%) were calculated by using the following equations:

(Bio-sorbed metal conc. (mg/kg) = Ci – Cf

Bio-sorption capacity % = Ci – Cf / Ci X 100

Where Ci =initial metal concentration and Cf = final metal concentration.

Sensory Evaluation

In order of providing valuable data for research on the factors that influence rice eating quality, Sensory evaluation of rice samples involved assessing the cooked rice's appearance, aroma, taste, and texture using human senses. Ten trained panelists evaluate these attributes to determine the overall eating quality and identify potential areas for improvement. Key sensory qualities assessed include adhesiveness, hardness, stickiness, dryness, whiteness, and aroma.

Scoring Systems: Panelists used a scale (1-5) to rate the intensity of each sensory attribute.

Statistical Method

All the analyses were assessed in triplicate, and the statistical evaluation of the data conducted using a one-way analysis of variance (ANOVA) (IBM Corporation, Armonk, NY, USA). To determine the significant differences among the treatment means, Duncan's multiple test range ($p < 0.05$) used.

Results and Discussion

The results of determination of Cadmium and Lead contents in the samples of studied rice in 2 groups of raw brown rice, *Oryza Sativa* L. (Hashemi-Tarom) samples without treating by bio-adsorbent were accomplished by FAAS, and reported as mean concentration in mg / kg in Table 1, all group had 5 subsamples and results are the mean of 5 replicates . All concentrations expressed as (mg/kg in Dry weight ± SE).

The maximum limit of Cadmium in rice has established about 0.06 mg/kg by Iran National Standard (No. 12968) and FAO set 0.2mg/kg, and Iran Standard (No. 12968) has set Pb intake limit of 0.15 mg/kg and WHO/FAO assigned permissible Pb intake limit of 0.3mg/kg respectively (Organization INS. 2013 ; ISIRI, 2010).

The mean contents of Pb and Cd in raw, rinsing, soaking by NaCl 2%, draining and cooking brown Hashemi-Tarom rice samples are shown in Figure 3 . It was discovered that rinsing 5 times of rice samples 5 times decreased heavy metals significantly ($p \leq 0.003$),

Table 1- Comparing Lead and Cadmium Contents in raw brown rice samples from two provinces: Gilan and Mazandaran

State	Pb content (mg/kg DW±SE)	Cd content (mg/kg DW±SE)	Ni content (mg/kg DW±SE)
Raw Brown Rice (M)	1.872±0.005	0.0965±0.0012	8.456±0.0002
Raw brown Rice (G)	1.003±0.004	0.0667±0.0022	7.564±0.0009
Mean	1.438±0.004	0.0812±0.0017	8.010 0.0006

M: Mazandaran Samples, G: Gilan Samples

SE: standard Error

The minimum and maximum Cd and Pb contents were 0.0041±0.0002 and 0.7321±0.0011 (mg/kg DW ±SE) in the farm in Gilan Province. The comparison of heavy metal contents in Table 1 , obviously, rice samples in Mazandaran province show higher cadmium and lead contamination, likely resulting from a combination of factors related to the region's environment and agricultural practices. These include the use of contaminated irrigation water, the application of certain fertilizers and pesticides, and the proximity of some rice paddies to industrial areas. The results in Table 1, revealed that heavy metal concentrations as contaminants differ significantly between Gilan and Mazandaran provinces ($p<0.05$), particularly in agricultural products like rice. These differences probably be attributed to variations in industrial activity, agricultural practices (including pesticide use), and natural geological factors.

Findings of current research also demonstrated that the mean content of Cadmium and Lead in the most samples from samples is higher than maximum levels set by national standard(ISIRI, 2010) and a few of them higher than maximum set by FAO/WHO.

The best adsorbent efficacy achieved with higher doses (5% and 10 percent) of biomass, but this negatively impacted sensory characteristics, making the adsorbent unsuitable for eating. The Cooked rice samples at higher doses were unsuitable for eating as exhibited a change in color and developed an off-putting. This trade-off between adsorption performance and sensory quality is a common challenge in the field. Therefore, authors eliminate the potential of removing heavy metals by the doses of 5 and 10 percent of Orange Pomace.

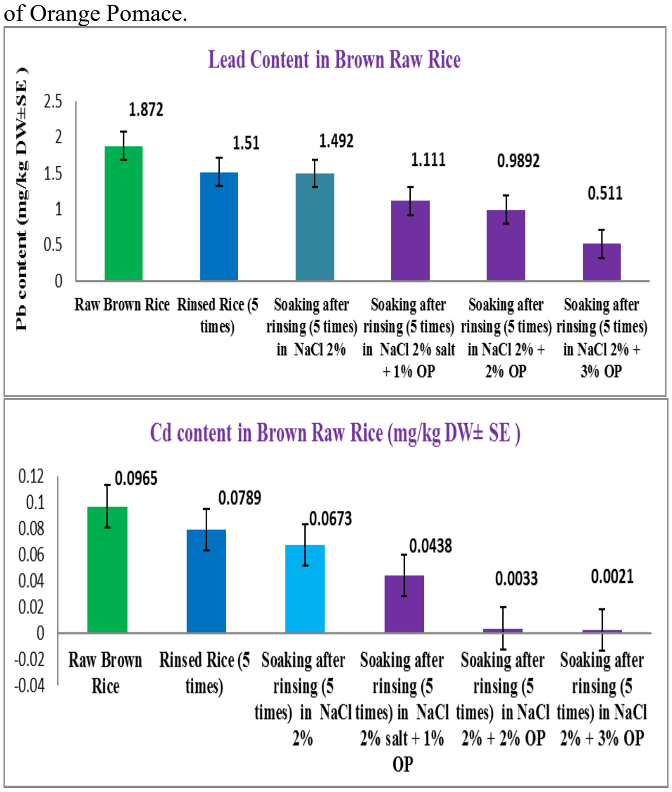

Figure 3- Lead and Cadmium Contents (mg/kg DW ±SE)in studied Brown Rice in different state of raw, rinsed and soaking in presence of NaCl 2% and different doses of OP (Orange Pomace) as bioadsorbent after 1 hour in room tempreture (25 ±1 °C)

*SE=Standard Error

The results from Figure 3 , sharply revealed that soaking rice in a salt solution or with a bio-adsorbent can significantly reduce lead and cadmium content. Studies have shown that soaking rice can significantly ($p< 0.005$) decrease the levels of these heavy metals, potentially rendering the rice safer for consumption.

Figure 4, highlights the beneficial role of bioadsorbents in reducing lead (Pb) and cadmium (Cd) levels in rice, making it safer for consumption. Bioadsorbents, materials that bind to and remove heavy metals, are shown to effectively lower the concentrations of these toxic metals in rice samples, rendering them suitable for human intake.

Current research indicates that orange pomace, a citrus fruit by-product, can effectively act as a biosorbent to remove heavy metals like lead and cadmium from contaminated brown rice. This utilization supports the principles of a circular economy by repurposing waste into a valuable resource for environmental remediation. The use of orange pomace offers a sustainable and cost-effective approach to both waste management and remediation of heavy metal contamination.

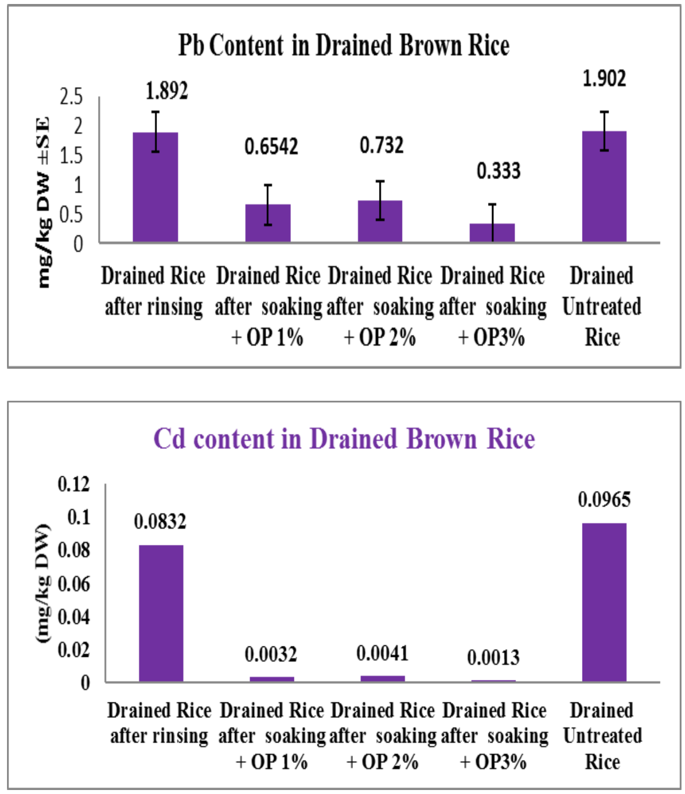

Figure 4- Lead and Cadmium Contents (mg/kg DW ±SE)in drained Brown Rice, after 1 hour soaking by NaCl 2% and different doses of OP (Orange Pomace) in comparison to untreated samples in room temperature (25 ±1 0C)

*SE=Standard Error

The results illustrated in Figure 4 revealed that orange pomace is an environmentally friendly adsorbent with a high capacity for significantly removing cadmium ($p \leq 0.001$) and lead ($p \leq 0.003$). Orange pomace, rich in cellulose and other bioactive compounds, can bind to heavy metal ions like lead and cadmium through a process called biosorption. The potential of orange pomace, particularly the peel, was explored through FTIR spectroscopy to identify functional groups, which can inform its valorization. Studies analyzing the Fourier Transform Infrared (FTIR) spectrum in Figure 5, of pomace powders reveal key functional groups associated with lignocellulosic materials, such as O-H, N-H, and C-O bonds. This information is crucial for understanding the material's properties and potential applications, including its use in food products, biofuel production, and as a precursor for activated carbon. The spectra obtained in absorbance mode from 4000 to 400 cm−1 a resolution was 0.44 cm−1.

Peaks at 2850 cm^{-1} and 2920 cm^{-1}: These peaks are associated with both symmetric and asymmetric stretching vibrations of C-H bonds, likely from alkanes and other organic molecules within the pomace. Peaks in the 1600-1580 cm^{-1} region:

These narrow peaks indicate the symmetric stretching of C=C bonds in aromatic rings, commonly found in lignin. Other peaks: Depending on the specific composition and treatment of the pomace, other peaks may be observed, such as those related to carboxyl groups (C=O stretching) or other functional groups

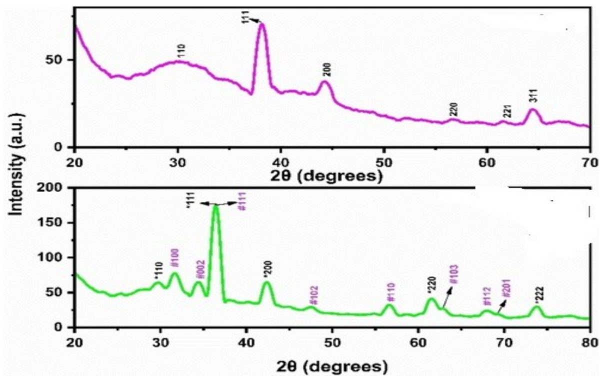

Figure 5- The FTIR of Orange pomace

Conclusion

The study investigated how well orange pomace, a byproduct of orange processing, could adsorb lead and Cadmium as harmful and hazardous heavy metals from Tarom-Hashemi brown rice. The research aimed to see if orange pomace could help reduce lead and cadmium contamination in the native cultivated rice in Iran. The results indicated that orange pomace, due to its pectin content and ability to bind with lead as well as cadmium and significantly assisted in adsorbing and detoxifying the heavy metal from the rice. Orange pomace, a citrus by-product, could be employed as a proper, low-cost, and available biosorbent to remove heavy metals from brown rice. This involves utilizing the orange peel's ability to bind with heavy metals, effectively reducing their presence in the rice.This process can be a cost-effective and environmentallyfriendly method for detoxifying rice contaminated with heavy metals.

5. Acknowledgment

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6. Conflict of interest

The authors declare that they have no conflicts of interest.

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Informed Consent Statement

Not applicable

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