





## Article

# Ultrasound- and Microwave-Assisted Extraction of Pectin from Apple Pomace and Its Effect on the Quality of Fruit Bars

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**Abstract:** The article investigates the process of pectin extraction using ultrasonic and microwave techniques from apple pomace generated during juice production in the context of circular bioeconomy. The extraction yield, equivalent mass, content of methoxyl groups, content of anhydrogalacturonic acid, and degree of esterification of pectin were investigated. These indicators varied depending on the parameters and extraction method. The resulting pectin displayed a co-extracted total polyphenol content (TPC) ranging from 2.16 to 13.05 mg GAE/g DW and a DPPH radical inhibition capacity of 4.32–18.86  $\mu\text{mol TE/g}$ . It was found that the antioxidant activity of raw pectin is correlated with TPC and with the content of terminal groups released during the polysaccharide degradation process. The extracted pectin was used as a binding and coating agent for dried fruit bars. Evaluation of water activity ( $a_w$ ), TPC and total flavonoid content (TFC), together with sensory and microbiological analyses of the fruit bars over a period of 360 days, revealed a protective effect of pectin: reducing moisture loss, minimizing the degradation of bioactive compounds during storage, and maintaining the potential antioxidant activity of the product.

**Keywords:** apple pectin; ultrasound; microwave; extraction; phenolic content; antioxidant effect; biopolymer coating; dried fruits; quality



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## 1. Introduction

Apples are some of the most widespread fruits, with multiple benefits for the consumer's health. In the Republic of Moldova, in 2017, apple production reached 430,000 tons from a total orchard area of 56,000 hectares. It was forecasted that the cultivated area for apple trees would increase by 52,400 hectares during the period of 2017–2027, resulting in a total apple production of 793,000 tons [1]. Significant amounts of apple pomace (globally, 4 million tons/year) are produced as a byproduct during the processing of apples for jams, juices, and fermented products. Despite the fact that apple pomace is primarily used as animal feed or fertilizer, it is a substantial source of functional components such as carbohydrates, dietary fibers (including pectin), phenolic compounds, and others [2]. The pectin derived from apple pomace is used in the pharmaceutical, food, cosmetic, and other industries, where it serves as a biopolymer, preservative, antioxidant, anticorrosive agent, protective agent for diverse surfaces, etc. [2,3]. Fibers obtained from fruits offer an advantage over cereal fibers due to their superior solubility, lower phytic acid content, and the presence of bioactive molecules associated with antioxidant activity [4]. Pectin is industrially obtained from apple pomace through conventional extraction (CE) methods, such as using hot acidified water with either mineral acids (sulfuric, hydrochloric, nitric) or organic acids (citric, malic, oxalic) from pH 1.5 to 3.0 and temperatures ranging

from 60 to 100 °C for 0.5 to 6 h, followed by alcoholic precipitation [2,5,6]. The cost-effectiveness and optimization of pectin extraction can be improved through application of unconventional extraction techniques such as the microwave-assisted extraction (MAE) [7], ultrasound-assisted extraction (UAE) [8,9], pulsed electric field extraction [10], subcritical water extraction [11], enzyme-assisted extraction [12], as well as combinations of different extraction methods [13–15]. The sustainability of unconventional methods such as UAE and MAE were proved, as the methods exhibit reduced energy and reagent consumption, shorter processing times (15–30 min as opposed to 1–3 h), and improved quality and yield of the final product compared to conventional methods [16–18].

Several studies have confirmed that the antioxidant activity (AA) of pectin is influenced by the structure and composition of its chains, as well as by the presence of co-extracted contaminants in the polysaccharide matrix, which are associated with polyphenols, proteins, and other antioxidants [19–22]. Apple pectin, depending on its concentration, exhibited an approximately 5-fold greater DPPH radical-scavenging effect compared to other polysaccharides [23].

The pectin extraction method also influences AA. Pectin obtained by unconventional methods from various sources, with lower degree of esterification (DE) and higher anhydrogalacturonic acid (AUA) content, exhibits a higher AA compared to pectin extracted through CE [11,24,25]. Wang et al. [26] reported that pectic polysaccharides extracted with hot-compressed water from apple pomace showed *in vitro* AA and an inhibitory effect on free radicals. The IC<sub>50</sub> values of such pectin oscillated between 1.4–3.5 mg/mL for 2,2-diphenyl-1-picrylhydrazyl (DPPH) scavenging and about 1 mg/mL for 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS).

Recent studies have shown that modified pectin obtained through unconventional methods displays different structural features [26–28]. It contains a higher amount of galactoside residues compared to xylan and arabinan [28] and shows more advanced radical-scavenging and anticancer activities compared to native pectin and pectin extracted through conventional methods [29,30]. Hydrolytic degradation of polysaccharides during unconventional extraction is accompanied by the formation of several reducing terminal groups, respectively, by the improvement of the antioxidant potential of pectin [29,30].

The DE of pectin also influences its AA due to its ability to chelate heavy metal ions [31,32]. A study on the influence of modified citrus pectin on the oxidative stability of flaxseed/sunflower emulsions confirmed that low-methoxyl pectin (DE of 33%) exhibited a higher lipid antioxidant potential compared to high-methoxyl pectin (DE of 58%) [33].

Multiple research studies have shown the positive influence of pectin on health status [2,3,6,27]. Pectin has probiotic properties and contributes to the proper functioning of the intestine, as it retains water and various waste substances, facilitating the elimination of toxins and protecting the colon's mucus membrane [31,34]. Pectin can bind and remove heavy metals from the body [31,32,34] and lower the cholesterol level [34,35] and serum glucose level [36]. Additionally, it has the capacity to capture free radicals and reduce the risk of cancer [37]. Pectic polysaccharides reduce inflammation, have antibacterial properties [38], and stimulate the immune response [39].

Pectin is used in the food industry as a thickening additive; it acts as a protective and stabilizing colloid in food and beverages. Pectin with a DE > 50% (high-methoxyl pectin—HM) forms a gel in solutions with a high concentration of sugar or solid substances, at a pH lower than 3.5. This is applied in the production of jams and jellies, fruit fillings, desserts, etc. [3]. Pectin with a DE less than 50% (low-methoxyl pectin—LM) forms a gel in a wide pH range (2.0–6.0), in the presence of calcium ions or other multivalent cations. It is used in the production of dietetic dairy products, soy-based products, etc. [40]. Other properties associated with pectin are: protein stabilization, softness in texture, increase in volume, and syneresis control in low-calorie foods [4,6,41].

Studies conducted in recent years have highlighted the sustainability of using pectin for the formulation and preservation of functional foods and encapsulation of bioactive compounds [42]. The production of edible coatings based on pectin and other biodegrad-

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able polymers is encouraged by the United States Environmental Protection Agency (US EPA) program to minimize packaging waste. Pectin-based nanoemulsions play an important role in creating a new generation of active packaging with health benefits [43]. Pectin-based films are biodegradable and possess excellent mechanical properties, providing the possibility to extend the shelf life of packaged foods [44,45], control moisture loss, and reduce the degradation of bioactive compounds during storage [46,47].

**The objective of this study** was to examine the impact of unconventional extraction methods, such as ultrasound-assisted extraction (UAE) and microwave-assisted extraction (MAE), on the yield, properties, and antioxidant activity of raw pectin extracted from apple pomace. The study also aimed to assess the potential of pectin as a binding and coating agent in the production of dried fruit bars; the protective effect of pectin films on functional products during storage was also investigated.

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