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REVIEW

Emerging techniques assisting nixtamalization products and by-products processing: an overview

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ABSTRACT
The production of worldwide nixtamalized products has increased in Latin American countries over the last years. For a better maize handling and exploitation of its nutritional elements, maize is subjected to a nixtamalization pretreatment protocol, which produces meaningful chemical, nutritional and quality changes in maize and its derived products, but large amounts of its primary by-product, well-known as 'nejayote', are also produced. Importantly, nejayote is usually discarded into the urbanized sewage with minimal treatment. Today, according to the recent research reports, new emerging techniques and protocols have been implemented to improve the nixtamalization products and by-products processing. New valorization approaches and biotechnological developments (including biotransformations) toward the reuse of nejayote have been developed according to its considerable content of biomolecules. Therefore, the goal of this paper is to provide a comprehensive review of the main development works at assisting nixtamalization products and by-products processing. Herein, particular attention is paid to experimental insights dealing with the valorization of nejayote.

KEYWORDS
Nixtamalization; waste valorization; emerging techniques; nixtamalized products; nejayote

Introduction

Nixtamalization is an ancient technique used by Latin American populations for tortillas making, instant corn flours, snacks and other traditional preparations. The industry of tortillas and nixtamalized snacks has been steadily increasing around the globe. Only in the U.S., the projected sales for tortillas approximate 3.659 million U.S. Dollars by 2023, whereas annual *per capita* intake in Mexico averages 79.5 kg (STATISTICA 2020).

The traditional process consists of an alkaline cooking of the whole grain using 0.1–0.2% $Ca(OH)_2$, followed by a steeping step ∼12 h and 2–3 washes to remove the pericarp and residual alkali (Argun and Argun 2018). Such cooking water, called nejayote, is discarded before the washing to eliminate soluble components and lime excess. Industrially, this practice generates an important amount of polluting waste with high alkalinity and chemical oxygen demand. It has been documented that the average production (in Mexico) of wastewaters by the nixtamalization process is about 1500–2000 m^3 per day, which means around 1.2 million m^3 per month and totally 14.4 million m^3 per year (Castro-Muñoz et al. 2017).

Moreover, along with nejayote, different corn elements are also discarded, including endosperm, germ, dietary fiber, and the pericarp. Within such elements, different types of biomolecules have been identified, such as arabinoxylans (Chen et al. 2019), phenolic compounds (e.g. ferulic, dehydrodiferulic, hydroxycinnamic, dehydrotriferulic and p-coumaric acids) (Díaz-Montes, Barragán-Huerta, and Yáñez-Fernández 2020), and carbohydrates (Díaz-Montes, Castro-Muñoz, and Yáñez-Fernández 2016), to mention just a few. Considering the importance of such molecules, the research community has started the exploration of nejayote as source of compounds with nutraceutical and pharmaceutical interest (Gutiérrez-Uribe et al. 2010).

Therefore, food technicians and scientists are today evaluating and developing new methods to reduce the negative environmental effect of nejayote, either by using emergent techniques that minimize water usage and waste streams or by re-valorating nejayote as source of valuable biomolecules.

Based on those scopes, the current review paper provides a comprehensive outlook about the ongoing research works focused on new emerging techniques and processing technologies toward nixtamalization products and valorization of its main by-product nejayote, as well as the most recent biotechnological breakthroughs. By exploring the data acquired mainly at lab-scale experiments, we have addressed in detail the most relevant approaches and insights in the field.

Nejayote

Nejayote is a wastewater of high alkalinity (pH > 10). The residual organic matter (mainly $CaCO_3$) along with the biological oxygen demand (BOD) and chemical oxygen demand (COD) make this pollutant effluent difficult to treat. For this reason, nejayote is discarded into the sewage without

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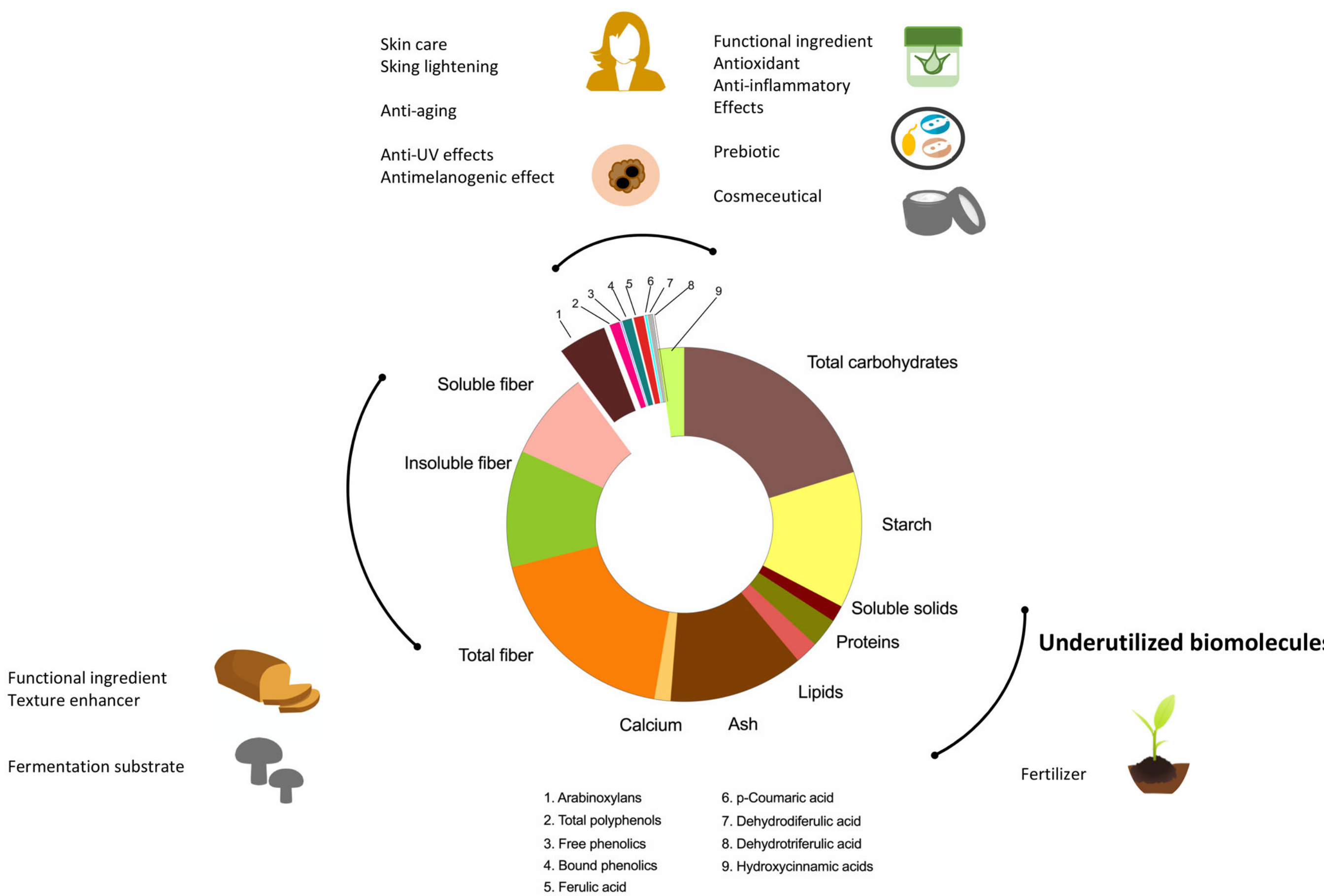

Figure 1. Distribution of valuable biomolecules of nejayote and their potential applications. Values are represented as percentage based on the information gathered from: (Acosta-Estrada et al. 2014, 2019; Argun and Argun 2018; Ayala-Soto et al. 2014; Campechano Carrera et al. 2012; Castro-Muñoz and Yáñez-Fernández 2015; Díaz-Montes, Castro-Muñoz, and Yáñez-Fernández 2016; García-Depraect et al. 2017; Gutiérrez-Uribe et al. 2010; Niño-Medina et al. 2009; Paz-Samaniego et al. 2015; Ramírez-Araujo, Gaytán-Martínez, and Reyes-Vega 2019; Téllez et al. 2016; Valderrama-Bravo et al. 2012; Villela-Castrejón, Acosta-Estrada, and Gutiérrez-Uribe 2017).

any treatment as a common practice among local tortilla producers (Gutiérrez-Uribe et al. 2010). Given the polluting nature of this wastewater, several attempts have been made to reduce the COD and BOD (Asaff Torres and Reyes Vidal 2014). Aerobic and anaerobic biological treatments have been applied to nejayote to transform the organic matter into biogas, although this process is mostly suitable for large industrial processes (Duran de Bazua 1987). Other purification methods for nejayote are multi-step treatments that include the enzymatic hydrolysis of solids, flocculation, micro-, ultra-, and nanofiltration/reverse osmosis that yield streams of enough quality that can be safely discarded (Asaff Torres and Reyes Vidal 2014). The enzymatic treatment with laccase in the presence of chitosan was also effective to achieve 70 and 78% reductions of COD (García-Zamora et al. 2015).

Despite its polluting nature, nejayote is also a potential source of residual solids of biological interest, including phytochemicals such as ferulic acid, arabinoxylans, carotenoids and some vitamins (Ayala-Soto et al. 2014; De La Parra, Serna Saldivar, and Liu 2007; Gutiérrez-Uribe et al. 2010; Niño-Medina et al. 2009). A growing number of studies have evaluated nejayote as a potential source of biomolecules, however, not enough information about the composition, bioaccessibility, bioavailability and bioactivity of these phytochemicals is available. More in-depth studies are needed to completely characterize this wastewater and to validate the technological processes techniques that may reduce nejayote generation, its alkalinity and recovery of biomolecules.

Nixtamalization and nejayote composition

Maize kernels suffer several physical and chemical changes during nixtamalization and steeping. During cooking and steeping of maize, starch is gelatinized, proteins become more available and various components of maize are leached into the cooking water. These compounds comprise soluble carbohydrates, dietary fiber, proteins and vitamins that are lost when nejayote is discarded. Some reviews have published the nejayote composition, however, most of them have not considered the complete profile of biomolecules. Figure 1 summarizes the average concentration of the main components of nejayote, including valuable phytochemicals and its potential use as functional ingredient. It is worth to note, that the nature and functionality of these phytochemicals are greatly dependent on the environmental conditions used for cultivation and the genotype of maize, these factors should be considered for their use in food or other applications.

The composition of nejayote is variable, about 3.2–14.5% comprises the dry matter; from which 10–30.1% is fiber, 6.4–32% starch, 1.3–8.9% proteins and 0.15–5.7% calcium (Campechano Carrera et al. 2012; Acosta-Estrada et al. 2014; Díaz-Montes, Castro-Muñoz, and Yáñez-Fernández 2016; Argun and Argun 2018; Ramírez-Araujo, Gaytán-Martínez, and Reyes-Vega 2019). Among this variable composition, high-added-value compounds are contained in nejayote effluents: 0.21–1.19 g of ferulic acid/100g dry matter (Acosta-Estrada et al. 2014; Villela-Castrejón, Acosta-Estrada, and Gutiérrez-Uribe 2017), 0.5–18.9% arabinoxylans (Ayala-Soto et al. 2014; Paz-Samaniego et al. 2015) and 1.04–36.6 µg/mg of total hydroxycinnamic acids (Villela-Castrejón, Acosta-Estrada, and Gutiérrez-Uribe 2017), from which, 0.25–0.75 µg/mg are attached to arabinoxylans (Ayala-Soto et al. 2014; Niño-Medina et al. 2009).

The physical and chemical phenomena occurring throughout the nixtamalization process, determine nejayote composition. First, pericarp undergoes hydrolysis and partial solubilization of certain components by the alkaline pH of the medium. The outermost waxy layer of the pericarp is partially removed during the initial stage of cooking, allowing the alkaline solution to diffuse inside the corn grain (Gutiérrez-Cortez et al. 2016). A further hydrolysis of ester bonds within the cellulose-hemicellulose-lignin structure allows the detachment and lixiviation of small amounts of cellulose and other non-cellulosic components, such as lignin, arabinoxylans and ferulic acid (Santiago-Ramos et al. 2018). Therefore, an important portion of the solids found in nejayote is composed of the fiber fractions, as shown in Figure 1.

The effect of alkali addition has not been investigated, although, there are certain differences in the fiber content and composition when traditional, ecological or commercial nixtamalization is used. In the traditional process the pericarp is removed, whereas in the ecological process the addition of calcium salts allows a greater retention of the pericarp, thus, a higher fiber content is expected in nejayote produced by the traditional process (Campechano Carrera et al. 2012).

An important portion of the non-cellulosic polysaccharides is comprised by arabinoxylans, linear α-(1,4)-D-xylopyranose polymers with L-arabino- furanose side chains. Significant amounts of feruloylated arabinoxylans have been found in nejayote (Paz-Samaniego et al. 2015), which represent a potential source of antioxidant fiber that can be recovered and used as functional ingredient (Castro-Muñoz et al. 2017).

Regarding phytochemicals, corn is a good source of ferulic acid, and anthocyanins in the case of pigmented corn varieties. Most of these compounds are lixiviated to nejayote in the cooking and steeping stages, for instance, up to 79% ferulic acid and 50% of anthocyanins are lost after nixtamalization (Cortés et al. 2006; Mora-Rochin et al. 2010).

The starch content of nejayote has been reported in few publications (Pflugfelder, Rooney, and Waniska 1988, Argun and Argun 2018), although its nature has not been determined. It would be interesting to study whether this compound could be a potential source of retrograded resistant starch or type-V amylose-lipid complexes. Numerous health benefits have been associated with resistant starch, including lower levels of blood lipids, satiety effects and gut microbiota improvement (Birt et al. 2013; Ramírez-Jiménez et al. 2015).

A remarkable characteristic of nixtamalized corn is its calcium content. From the nutritional point of view, nixtamalized products represent a major source of calcium for many Latin American populations, especially in the rural areas (Willows 2016). Not only the higher content of this mineral is remarkable, but also the increased bioavailability reached during nixtamalization. A study conducted with Mexican women who consumed nixtamalized tortillas showed a 25-fold increase in calcium absorption compared with those who consumed non-nixtamalized tortillas (Rosado et al. 2005). Nejayote contains up to 5.7% of this mineral, that might improve the nutritional profile of products enriched with nejayote solids (Acosta-Estrada et al. 2014). Further studies are needed to address the bioavailability and bioactivity of residual calcium to explore a feasible application as functional ingredient. It is worth to note, that the ash content is higher in the ecological process than in the traditional one. This parameter is associated with the residual calcium content in nejayote, thus, at higher ash concentrations, less calcium is lixiviated to the wastewater.

Potential applications of biomolecules found in nejayote

Functional ingredients and cosmeceuticals

The feruloylated arabinoxylans (AX) found in nejayote are considered valuable phytochemicals and functional ingredients. The gum-like properties of AX are exploited in the food industry, since they are used as enhancers of texture and viscoelastic properties of doughs; as emulsifiers in breads and sausages, and as hydrocolloids and fat replacers (Ayala-Soto, Serna-Saldívar, and Welti-Chanes 2017; Herrera-Balandrano et al. 2019).

AXs are considered dietary fiber; about 85% of these compounds are comprised by soluble fiber, whereas insoluble fiber is present in very low values. It has been reported that AXs from corn bran exhibit antioxidant capacity due to the ferulic acid moieties attached to these polymers (Herrera-Balandrano et al. 2020). Given the viscosity of AXs, these compounds have been used for the controlled delivery of insulin and β-lactoglobulin targeting diabetes and the colon microbiota, respectively (Berlanga-Reyes et al. 2009). Furthermore, a recent study showed that AXs isolated from nejayote have prebiotic potential since they influence the microbiota composition; specially AXs with low degree of polymerization have demonstrated positive effects on the growth of *Bifidobacteria* (Chen et al. 2019). Given the antioxidant and immunomodulatory effects observed for AXs in general, the anticancer activity of these compounds has also been investigated using bran from other cereals, therefore, AXs from nejayote might also have this potential benefit.

The physico-chemical characteristics of AXs resemble those of β-glucans, specially the viscosity and soluble fiber-like behavior, which give a hint about the variety of potential bioactivities they may have. Thus, the functional effects of AXs might not be limited to the food industry, other interesting bioactivities should also be explored.

Among the hydroxycinnamic acids identified in nejayote (p-coumaric and ferulic acid), ferulic acid (FA) is a 4-hydroxy-3-methoxycinnamic acid considered a potent antioxidant with diverse applications in the food and cosmetic industry. This compound is lixiviated to nejayote during the washing steps when pericarp is removed. The amount of FA contained in nejayote is remarkably higher, about 125 times more FA was found in nejayote compared with the nixtamalized product (Argun and Argun 2018).

FA recovered from nejayote has been used as an ingredient to enrich bread, increasing its antioxidant capacity by 70% (Acosta-Estrada et al. 2014). Other uses of this phytochemical include: antioxidant, antidiabetic and anticancer applications, food preservative and ingredient for cosmetics (Kumar and Pruthi 2014; Panwar et al. 2018).

Moreover, ferulic acid has been applied in skin care formulations for anti-wrinkling and antioxidant purposes (Taofiq et al. 2015). It has been demonstrated that FA has a significant capacity to absorb UV radiation, acting as a topical photoprotective agent (Saija et al. 2000). FA has shown to reduce UVB-induced erythema (Murray et al. 2008) due to a reduction in the expression of matrix metalloproteinases that contribute to photoaging and skin cancer development (Staniforth et al. 2012). In this regard, a transdermal hydrogel patch containing FA has been developed to treat skin damage caused by UV radiation (Bai et al. 2014). FA has also been used in face masks, antioxidant and moisturizing products (Kumar and Pruthi 2014). Since nejayote is a source of ferulic acid with known antioxidant capacity, there is an opportunity for the recovery of these compounds and their further application in the health and cosmetic sector.

Within the biomolecules found in nejayote, FA is the most studied and promising compound. The bioaccessibility and absorption of FA isolated from nejayote through the intestine have been previously explored (Villela-Castrejón, Acosta-Estrada, and Gutiérrez-Uribe 2017); important features if the intended use is for oral intake. Skin permeation and the local topical action of FA have also been proven in different formulations using in vitro and animal models. Yet, pharmacological and toxicity studies are needed for FA recovered from nejayote, given the alkalinity of the wastewater.

Similarly, p-coumaric acid (PA) not only exhibits antioxidant, anti-inflammatory and anticancer activity, but functionality for cosmetic formulations. PA has shown to be effective as dermatological agent to decrease hyperpigmentation. PA inhibits the enzyme tyrosinase by competitive displacement, which in turn, causes a reduction in melanin synthesis and reduces skin pigmentation. Other potential uses of PA include protection against UV, antimelanogenic activity and oxidative stress reduction. Regarding pharmacokinetics, PA is absorbed in the small intestine and its bioavailability has been demonstrated measuring PA concentrations in blood.

As a whole, nejayote has been spray-dried and microencapsulated to assess its functionality. Nejayote powder obtained by spray-drying was used as substrate for solid-state fermentation with different fungi strains (Acosta-Estrada et al. 2019); a significant increase of the phenolic content (31–325%) and soluble fiber (45.49%) was observed after fermentation with specific strains. This treatment does not require special conditions or expensive equipment; thus, it can be implemented industrially to recover a nejayote by-product with enhanced nutritional and phytochemical profile. Moreover, this process can be coupled to additional operations to ensure the delivery of the biomolecules. For instance, microencapsulation has shown to be an effective method for the controlled delivery of phenolics from nejayote (Villela-Castrejón, Acosta-Estrada, and Gutiérrez-Uribe 2017). With this method, a 100% release of total phenols was achieved during a simulated gastro-intestinal digestion using different encapsulating agents.

Given the potential functionality of the biomolecules contained in nejayote, some physical-separation techniques, biotechnological approaches and other environmentally friendly process are used to recover phytochemicals from nejayote. These procedures will be detailed in section 6.

Emerging techniques used to minimize nejayote production

Given the environmental impact of wastewater produced by the nixtamalization process, alternative technologies have been proposed to avoid or minimize nejayote generation. Emergent methods such as ohmic heating, extrusion, microwaving, infrared and ultrasound technologies have aroused as feasible solutions to this issue. Table 1 describes the main operation features of each technology, as well as their impact on nejayote production and some valuable biomolecules.

Ohmic heating

Ohmic heating (OH) is an electro thermal technology used for the efficient and rapid cooking of foods. This technology is based on the application of an electrical current to a food, which generates internal heat due to the fooďs resistance to this electrical stimulus (Varghese et al. 2014). With this technology, foods are cooked uniformly, which in turn, reduces the energy expenditure and processing time. It is worth to note that no wastewater is generated with this process (Ramírez-Jiménez et al. 2019).

Since the amount of heat generated is related to the current and voltage applied, some variables must be controlled to achieve an optimal product. Water, particle size, viscosity and thermal conductivity of the material should be considered (Gaytán-Martínez et al. 2012).

A method for producing corn flours was developed using continuous ohmic heating (Gaytán-Martínez et al. 2012; Ménera-López et al. 2013; Morales-Sánchez, Figueroa-Cardenas, and Gaytan-Martinez 2010). This method comprises three steps: (a) milling of raw corn to a 0.88 mm particle size, (b) addition of 0.3% lime and enough water to achieve 50% moisture, (c) nixtamalization

Table 1. Main processing conditions, nejayote production and valuable biomolecules in wastewaters of different technological processes.

Technology	Processing conditions	Main nutritional features	Nejayote production	Valuable biomolecules	References:
Traditional nixtamalization	Boiling cooking: 25–45 min $Ca(OH)_2$: 0.1–2% Steeping time: 12–20 h	Higher availability of proteins. Gelatinization of carbohydrates. Amylose-lipids interactions (type V resistant starch)	0.4–0.7 L/Kg	Soluble and insoluble fiber, phenolic compounds and arabinoxylans	(Mariscal-Moreno et al. 2019; Ramírez-Araujo, Gaytán-Martínez, and Reyes-Vega 2019; Ramírez-Jiménez et al. 2019; Santiago-Ramos et al. 2018; Serna-Saldívar et al. 2013)
Ohmic heating165:91	Continuous process. Voltage applied: 120–130 VACS, 60 Hz, 1–2 min Temperature: 70–85 °C $Ca(OH)_2$: 0.3% Steeping time: 30 min pre-conditioning	Nutritional content similar than that of traditionally nixtamalized flours and tortillas. Bioavailability not evaluated. Preservation of insoluble and soluble fiber, phenolic compounds, mainly ferulic acid.	No wastewater produced.	NA	(Gaytán-Martínez et al. 2012; Ménera-López et al. 2013; Ramírez-Araujo, Gaytán-Martínez, and Reyes-Vega 2019; Ramírez-Jiménez et al. 2019)
Extrusion	Continuous process Temperature: 80–150 °C Moisture: 20–50% $Ca(OH)_2$: 0.2–0.3%	Gelatinization of carbohydrates. Dextrins formation Retrograded resistant starch Relatively high retention of phenolic compounds ∼75%	No wastewater produced.	NA	(Enríquez-Castro et al. 2020; Enríquez Castro et al. 2018; Escalante-Aburto et al. 2020; Gaxiola-Cuevas et al. 2017; Gómez-Aldapa et al. 1999; Martínez-Flores et al. 2002; Ramírez-Araujo, Gaytán-Martínez, and Reyes-Vega 2019)
Low-shear extrusion	Continuous process, 1–2 min Temperature: 70–85 °C Moisture: 45–65% $Ca(OH)_2$: 0.05–0.65%	Low starch dextrinization, controlled gelatinization.	No wastewater produced.	NA	(Contreras-Jiménez et al. 2014; Mercado Pedraza et al. 2014)
Microwaving	Batch process: 800–1650 W, 2450 MHz, 4–20 min $Ca(OH)_2$: 0.125–0.5%. Water addition: 1:1 proportion Steeping time: maximum 3.5 h	Nutritional content similar than that of traditionally nixtamalized flours and tortillas. Higher dietary fiber, lysin and tryptophan content. Reduction of aflatoxins.	6.2–8.34 mL/ L	NR	(Martínez-Bustos et al. 2000; Méndez-Albores et al. 2012; Pérez-Flores, Moreno-Martínez, and Méndez-Albores 2011)
Infrared	Batch process: 1300 W, 60 Hz Temperature: 80 °C Water addition: 3 L/Kg $Ca(OH)_2$: 1%. Cooking time: 37 min Steeping time: 18 h	Nutritional content similar than that of traditionally nixtamalized flours and tortillas. Higher yield than traditional and ultrasound processing	∼ 2.5 L/Kg plus 3 L/Kg of washing water	Soluble and insoluble fiber, phenolic compounds and arabinoxylans	(Méndez-Albores et al. 2014)
Ultrasound	Batch process: 550W, 24 kHz Temperature: 80 °C Water addition: 3 L/Kg $Ca(OH)_2$: 1%. Cooking time: 37 min Steeping time: 18 h	Nutritional content similar than that of traditionally nixtamalized flours and tortillas.	NR	Soluble and insoluble fiber, phenolic compounds and arabinoxylans	(Méndez-Albores et al. 2014)

NR – Not reported, NA – does not apply.

of the pre-conditioned raw flour in the continuous ohmic cooker, (d) dehydration and milling of nixtamalized flours.

Instant corn flours obtained by this process have shown adequate physico-chemical characteristics for tortilla making (Ménera-López et al. 2013), as well as a nutritional profile similar to that of corn flours obtained by the traditional nixtamalization process (Gaytán-Martínez et al. 2012). An important feature of this process is the lack of polluting effluents since no excess water is added to the raw mixture, moreover, less nutrients and phytochemicals are lost (Ramírez-Jiménez et al. 2019; Varghese et al. 2014).

A comparison study between the OH technology and the traditional process showed that the dietary fiber content is higher in flour obtained by OH because the pericarp is not removed (Ramírez-Jiménez et al. 2019). Phenolic compounds are important phytochemicals with known antioxidant properties. In this study, bound phenolics and total flavonoids were higher in corn flours obtained with OH, bound ferulic acid losses were negligible, whereas the traditional process causes losses up to 50%. OH seems to have an effect on the release of phenolic compounds from corn cell wall polysaccharides, increasing the free forms of these phytochemicals (El Darra et al. 2013; Ramírez-Jiménez et al. 2019).

Extrusion

Extrusion is widely used to obtaining different food products. This high temperature short-time (HTST) process is performed under a broad range of moisture (14-40%) and temperatures (70-140 °C). Several studies report the use of this technology for corn nixtamalization (Aguayo-Rojas et al. 2012; Arámbula et al. 1999; Contreras-Jiménez et al. 2017; Contreras-Jiménez et al. 2014; Enríquez Castro et al. 2018; Gaxiola-Cuevas et al. 2017; Mensah-Agyapong and Horner 1992; Platt-Lucero et al. 2010; Reyes-Moreno et al. 2013; Sánchez-Madrigal et al. 2014). The first experiments with extrusion added hydrocolloids and texture enhancers due to poor sensory properties of tortillas (Arámbula et al. 1999; Martínez-Flores et al. 1998; Platt-Lucero et al. 2010). During extrusion, starch undergoes excessive dextrinization and gelatinization due to a greater damage to its structure, which in turn, affects the water uptake capacity, rheology and viscosity of masas (Mercado Pedraza et al. 2014; Platt-Lucero et al. 2010). In this regard, processing conditions are a key factor to obtain flours and tortillas with good textural quality (Enríquez-Castro et al. 2020) as well as the type of extruder used. Longer pre-conditioning times before extrusion, the particle size of corn and the lime concentration have proven to significantly improve the rheological characteristics of nixtamalized corn and masas (Contreras-Jiménez et al. 2017). Moreover, a low-shear extrusion system was successfully used for the production of nixtamalized corn flours with lower starch damage and dextrinization, which improved the quality parameters of tortillas (Mercado Pedraza et al. 2014).

Extrusion has also been used to produce pre-gelatinized flours (instant flours) for tortilla making (Aguayo-Rojas et al. 2012). However, nearly 50% of the total phytochemicals (mainly phenolic compounds) are lost during the extrusion cooking of corn. Nevertheless, nixtamalized flours and tortillas produced by extrusion, retain more phenolic compounds and their antioxidant capacity is higher than that found in traditionally nixtamalized products (Gaxiola-Cuevas et al. 2017). An important feature of extrusion cooking is the reduction of water consumption and the lack of effluents.

Calcium salts and two steps nixtamalization

The use of calcium salts to replace lime in the nixtamalization process aims to decrease the pH and polluting power of nejayote. This so-called ecological nixtamalization uses calcium chloride, calcium sulfate and calcium carbonate. Mainly, calcium sulfate has achieved the highest pH reduction (Escalante-Aburto et al. 2020; Figueroa-Cárdenas, Rodriguez-Chong, and Veles-Medina 2011). As a consequence of the lower pH reached with the ecological nixtamalization (∼11–12), the pericarp is not completely hydrolyzed, preserving fiber, resistant starch and other nutrients (Bello-Perez et al. 2014; Campechano Carrera et al. 2012; Santiago-Ramos et al. 2015).

A variant in this process is the two-steps nixtamalization method that includes the use of calcium chloride and calcium lactate to improve the sensory and quality value of nixtamalized products. In this method, two 30-minutes cooking steps are performed, the first one with lime and the second one with calcium chloride or calcium lactate to decrease the pH of the final wastewater (Ruiz-Gutiérrez et al. 2012). Although the steeping time is reduced to 3 h, effluents are generated in every step of the process.

Other technologies

Microwave-assisted nixtamalization offers the advantage of short times of processing and low effluents generation. Typical processing times vary between 4 and 20 min applying 800–1650 W at 2450 MHz (Martínez-Bustos et al. 2000; Méndez-Albores et al. 2012; Pérez-Flores, Moreno-Martínez, and Méndez-Albores 2011); under these conditions, the proximate composition of nixtamalized flours remains practically unchanged, whereas the fiber content, lysin and tryptophan increase (Martínez-Bustos et al. 2000; Méndez-Albores et al. 2012). A substantial change in the quality parameters of flours and tortillas are observed with this technology.

The infrared technology has been used in few studies to obtain nixtamalized flours (Méndez-Albores et al. 2014; Zavala-Franco et al. 2016). This process has been useful to reduce aflatoxins and to increase yield of masa and tortillas. On the other hand, ultrasound-assisted nixtamalization has been effective in shortening cooking times for pozole elaboration and in controlling the extent of gelatinization in nixtamalization of corn flours (Méndez-Albores et al. 2014; Robles-Ozuna et al. 2016).

Alternative pathways for the treatment of nixtamalization by-products

Physical-separation techniques

As the primary by-product of this corn pretreatment, nejayote is generally rich in several classes of high-added value compounds, such as carbohydrates, fibers, phenolic compounds, sugars, calcium, etc. Among all these compounds, AXs are also valuable compounds contained in the by-product, and their recovery has been already explored. For intance, Niño-Medina et al. (2009) recovered feruloylated arabinoxylans ($\sim$0.23 μg mg^{-1}) by alkaline hydrolisis. Particularly, such a study reported that the high gelling ability of these compounds was attributed to the arabinose residues, which are esters linked on (O)-5 to ferulic acid (3-methoxy, 4 hydroxy cinnamic acid). At this point, AXs were able to form covalent gels by oxidation of FA, resulting in the creation of dimers (di-ferulic acid) and trimers (tri-ferulic acid) of ferulic acid, as covalent cross-linking structures (Paz-Samaniego et al. 2015). Moreover, the authors also stated that the recovery of this gum from a low-value maize waste represents a potential alternative over commercial gums used in the food industries, e.g. baking industry (Niño-Medina et al. 2009). Interestingly, it is important to point out that 30–40% of the total weight of the pericarp is based on AXs (Doner, Johnston, and Singh 2001). Herein, the pericarp, being complete or partially hydrolyzed in nejayote, could be recognized as a promising source of AXs. Importanly, the biodegradable films based on AXs represent a sustainable alternative toward the replacement of films manufactured from chemically synthetized polymers (Ghavidel et al. 2013; Ounkaew et al. 2018). In fact, it has been stated an increasing demand for the use of biopolymers (Castro-Muñoz and González-Valdez 2019; Galiano et al. 2018). For instance, based on the origin of AXs, there is a great potential in fabricating AX-based edible films for the preservation of perishable food (such as vegetables, fruits, among others).

Due to the potentiality of the compounds contained in nejayote, several authors have started the exploration and thus the extraction of such compounds using different technologies. For example, membrane-based technologies are today identified as emerging techniques for separating different categories of molecules from wastes (Castro-Muñoz et al. 2020). In principle, micro (MF), ultra (UF), and nanofiltration (NF) have been the main techniques implemented for the production of nutraceuticals from agricultural by-products (Castro-Muñoz and Fíla 2018; Castro-Muñoz, Barragán-Huerta, et al. 2016). The usage of such processes is becoming attractive in recovering phenolic compounds from diverse food by-products, including orange press liquor (Conidi, Cassano, and Drioli 2012), olive mill wastewaters (Galanakis et al. 2018), artichoke wastewaters (Cassano et al. 2015), winery-effluents (Teixeira et al. 2014), among others. In general, the design and implementation of multiple membrane operations in sequence, so-called “Integrated membrane processes” has allowed the fractionation and successful extraction of polyphenols. Such breakthrough was firstly developed in Italy (Conidi et al. 2015; Conidi and Cassano 2015), in which the primary goal was to reduce the occurrence of fouling phenomena in the subsequent membrane operations by prepending high pore size membranes, while the second objective was the fractionation of the extracts. Very recently, such approach of integrated membrane process was used for the first time for the valorization and fractionation of nejayote; Figure 2 illustrates the process design used.

Such a study implied the prepending of three-membrane steps. Firstly, the nejayote was subjected to pre-clarification using a MF (polysulfone, pore size 0.2 μm) step to remove the suspended solids and potentially reduce the organic matter. The MF supernatant (i.e. permeate) was used as the feed stream for the second step, which comprised the use of a UF (polysulfone, molecular weight cutoff 100 kDa) step. This particular stage aimed at the separation of carbohydrates (Castro-Muñoz, Cerón-Montes, et al. 2015), and the production of a clarified extract enriched in phenolic compounds and calcium. At this point, it is worth mentioning that the retentate of the UF 100 kDa step produced retentate samples containing up to 7 mg mL^{-1} (i.e. 7000 mg L^{-1}) of carbohydrates, which may be likely AXs. Such concentration of carbohydrates represents a huge quantity of biopolymer since few amounts of polymer (<5 wt.% polymer concentration) is needed for the preparation of edible films (Homez-Jara et al. 2018; Xiao, Tong, and Lim 2012). As final processing stage, the resulting UF permeate was used as a feed stream for the final tight UF (polysulfone, molecular weight cutoff 1 kDa) step to remove out the calcium molecules (Castro-Muñoz et al. 2020). The resulting permeate contained high phenolic content of 951 mg L^{-1} (in terms of FA) from an initial content of 1190 mg L^{-1}, which corresponds to the fraction with low total organic carbon content (Castro-Muñoz and Yáñez-Fernández 2015). Furthermore, the final permeate enriched in phenolic compounds had a considerable antioxidant activity (Castro-Muñoz, Barragán-Huerta, et al. 2016); which has been fully associated with phenolics (Gutiérrez-Uribe et al. 2010). It is important to note that the success of this integrated membrane process to separate the phenolic fraction has been attributed to the specific features of the membranes, such as pore size, membrane material, membrane configuration and structure (Castro-Muñoz, Conidi, et al. 2019). Typically, the asymmetric structure of the membranes allowed to reject compounds of lower molecular weight. According to Galanakis (2015), the asymmetric characteristic of membranes allows to consider the tight UF membranes (in the range of 1–2 kDa) as the border of nanofiltration. The membrane material also plays an important role for the separation. For instance, polysulfone, as a hydrophobic material, commonly repels water and molecules with a high affinity to water, improving the efficiency retention and recovery of membranes. Whereas, separation of polyphenols depends primarily on the membrane' narrow pore size due to the molecular sieving separation. The solutes' nature is also crucial. For example, phenolic compounds possess aromatic rings and aliphatic chains providing a hydrophobic nature to the whole

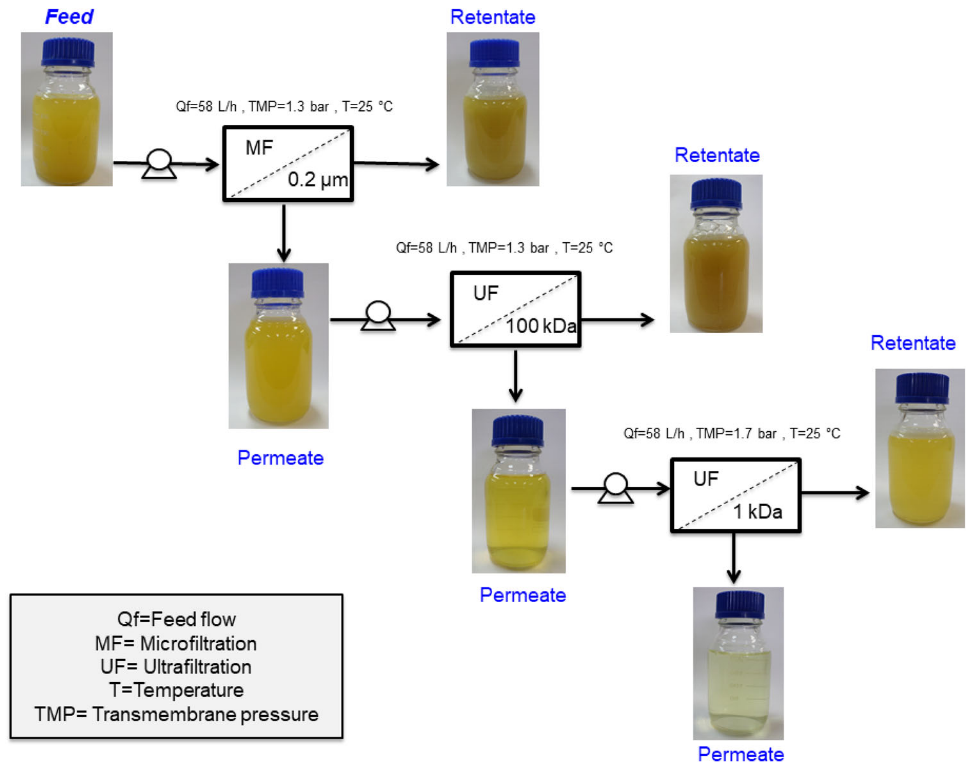

Figure 2. Integrated membrane system used for the valorization of nejayote (Castro-Muñoz and Yáñez-Fernández 2015).

molecule, this actually reduces the permeation performance of membranes (Castro-Muñoz et al. 2020; Galanakis 2015). According to some literature reports (Crespo and Brazinha 2010; Galanakis 2015), electrostatic and Coulombic interactions between membrane surface and solutes take place, which may improve the selective properties of the membranes (Castro-Muñoz et al. 2020). Regardless of the relevant findings obtained by fractionating nejayote (Castro-Muñoz and Yáñez-Fernández 2015), the process still has important drawbacks due to the high membrane fouling indexes (in the range of 22–87%), which will definitely cause operational issues in long-term processing. In fact, the fouling phenomenon has been recognized as the main bottleneck of pressure-driven membrane processes, that affects the performance in terms of permeate flux and rejection of membranes (Pichardo-Romero et al. 2020). In this way, an improvement in the design of the integrated membrane process should be implemented to decrease fouling, such as decantation, centrifugation, among others (Balderas-López et al. 2019; Ochando-Pulido and Martínez-Férez 2017). Interestingly, the initial permeability properties of the membranes were almost recovered by means of alkaline or enzymatic cleaning protocols (Al-Amoudi and Lovitt 2007). Such polysulfone membranes displayed cleaning efficiencies of about 99%, in other words, the initial water permeabilities were almost recovered (Castro-Muñoz and Yáñez-Fernández 2015). Very recently, the separation performance of an integrated MF-UF100 membrane process was evaluated at recovering polyphenols from three different maize types (Díaz-Montes, Barragán-Huerta, and Yáñez-Fernández 2020). It was found that the highest content of total polyphenols was contained in the fractions of UF100-Permeate, e.g. around 768, 800 and 776 mg GAE L^{-1} for white maize, red maize and purple maize, respectively. Such fractions presented an antioxidant activity of about 0.43, 0.33 and 0.33 TE mL^{-1}, respectively. HPLC analysis, revealed that the main hydroxycinnamic acids recovered at the end of the process (i.e. MF-UF100-UF1) were ferulic and *p*-coumaric acids, mainly in the fractions of UF1-Permeate in concentration of 99, 138 and 211 mg L^{-1} for white maize, red maize and purple maize, respectively (Díaz-Montes, Barragán-Huerta, and Yáñez-Fernández 2020).

Bio-transformation approaches

Since other types of biologically active matter can be found in this by-product, several biotechnological approaches have been proposed. For instance, Sanchez-Gonzalez et al. (2011) performed the isolation and characterization of two new alkaliphilic microorganisms contained in nejayote. Such unreported strains (NJY2 and NJY4) were identified as *Bacillus flexus*, which display interesting physiological properties, as well as the ability to produce specific molecules from agro-food wastes. As an example, the isolated NY2 strain can produce FA, and its ethyl and methyl-esters, from nixtamalized corn bran. Indeed, this approach was the first study in the framework of isolation of alkaliphilic microorganisms from nejayote, and the first case of phenolic acid esterases synthesized by alkaliphiles. These discovered alkaliphilic microorganisms may have a great potential within the treatment and transformation of tortilla manufacture residues (Sanchez-Gonzalez et al. 2011).

Since couple of years ago, nejayote has begun to be exploited toward specific biotechnological approaches, such as a new feedstock in fermentations (Ramírez-Romero, Reyes-Velazquez, and Cruz-Guerrero 2013; Salazar-Magallon et al. 2015). Particularly, Ramírez-Romero, Reyes-Velazquez, and Cruz-Guerrero (2013) analyzed the feasibility of nejayote as growth medium for probiotic bacteria and the production of bacteriocins. In this study, three different strains were evaluated (e.g. *Lactobacillus helveticus* IMAU70129, *Lactobacillus casei* IMAU60214, and *Lactobacillus rhamnosus* GG). The probiotic strains revealed a meaningful consumption of several components, such as reducing sugars (∼60–65%), total sugars (∼6–30%), and protein (∼1–15%). The resulting supernatant derived from fermentations inhibited about 10 and 14% for *Escherichia coli* K-12 and *Listeria innocua*, respectively. Afterwards, the authors stated that nejayote could be suitable to produce probiotics and bacteriocins, representing a low-cost feedstock.

FA isolated from nejayote can be an alternative in manufacturing specific ingredients for the food and beverage industry. For instance, FA can be converted into vanillic acid by fungi, this can be reached via a propenoic chain degradation to vanillic acid, a subsequent reduction reaction may result in the production of vanillin and vanillyl alcohol (Mathew and Abraham 2006). Here, introducing natural vanillin from nejayote to the market implies two relevant advantages, (i) absence of side effects through its consumption, and (ii) it is 1.3 times cheaper and faster to obtain than the natural vanillin from the vanilla pod processing (Castro-Muñoz et al. 2017).

Microencapsulation of nejayote has been recently carried out by Villela-Castrejón, Acosta-Estrada, and Gutiérrez-Uribe (2017). In principle, the microencapsulation deals with the production of tiny particles (in the order of microscale) surrounded by a coating wall material. Generally, it is used to embed, and thus, protect enzymes and food ingredients, among other biomolecules (Castro-Muñoz, Cerón-Montes, et al. 2015; Castro-Muñoz and Nieves-Segura 2018; Saénz et al. 2009). In Villela's study, the extract was encapsulated using maltodextrin (MD) or 2-hydroxypropyl-beta-cyclodextrin (HBCD) as wall materials. The resulting microcapsules were proposed as a delivery system of phytochemicals under simulated gastrointestinal digestion, rendering a complete bioaccessibility and release (100%) of the compounds under set conditions.

Environmentally friendly techniques for the treatment of nejayote

Within the exploration of re-using nejayote, some authors have profited on the extract for biotechnological and environmentally sustainable breakthroughs. Dominguez-Hernandez and coworkers utilized the by-product and ovine manure as fertilizers in maize production, aiming at the mitigation of the environmental impact of such agro-food wastes (Dominguez-Hernandez et al. 2020). The authors found out that nejayote-manure mixtures were 19% more energy efficient and provided 12% higher yield than chemical fertilization (including urea, ammonium diphosphate and potassium chloride), thus generating a benefit-to-cost ratio of 6.3. Such proposals were an alternative for waste treatments and also produced greater benefits than chemical fertilizers. In addition to this, the water recovered from nejayote was enough to give a 7.5 or 15 mm of gross irrigation over a crop cycle. In light of water recovering, membrane-based operations, by prepending according to their pore size, give the opportunity of obtaining process water for downstream tasks within the industries (Castro-Muñoz et al. 2017; Castro-Muñoz and Yáñez-Fernández 2015). At this point, in order to reach the physicochemical requirements of process water, it is likely that membranes with a narrower pore size (in the order of nanofiltration) will be preferred.

The use of nejayote as a raw substrate for the production of renewable energy sources, such as methane and biohydrogen (España-Gamboa et al. 2018; García-Depraect et al. 2017) has also been explored. Importantly, world government and research institutions are making meaningful efforts to promote the production of sustainable clean energy and mitigate environmental issues caused by the usage of fossil fuels. Therefore, researchers are looking for new sources for the possible manufacture of renewable energies (Panwar, Kaushik, and Kothari 2011). For instance, in Mexico España-Gamboa et al. (2018) proposed nejayote for methane production using a coupled system, which implied an anaerobic-packed column reactor and an up-flow anaerobic sludge blanket (APCR-UASB) process. This study was synergistically designed in order to: i) optimize the acidogenic phase by means of the UASB, and ii) to improve the methanogenic process. Insights indicated that a 19-day treatment concurrently provided a 96% chemical oxygen demand (COD) removal and produced biogas containing 84% methane. The methane yield was of about 282 L kg^{-1} of removed COD. On the other hand, García-Depraect et al. (2017) evaluated the co-digestion of vinasse and nejayote as complex raw substrates for the production of biohydrogen. Basically, optimum hydrogen production rate of 107 NmL H_2 Lr^{-1} h^{-1}, which means normalized volume (mL) bioH_2 per liter of bioreactor and hour at standard temperature and pressure (0 °C, 1 atm), as well as hydrogen yield of 115 NmL H_2 g total volatile solids^{-1} (TVS^{-1}) were reached at a vinasse/nejayote ratio of 80/20. The authors stated that this achievement was attributed to the synergistic effect of vinasse and nejayote possessing nitrogen, iron, magnesium, phosphorus and alkalinity. Following similar principles, further optimization and potentialities of nejayote are currently the scope of the study for biohydrogen production (García-Depraect, Rene, et al. 2019; García-Depraect, Valdez-Vázquez, et al. 2019).

Concluding remarks and future directions

This paper has reviewed the most used pathways, applications and approaches toward green and efficient technologies in processing nixtamalization products (such as ohmic heating, extrusion) and by-products. The main changes in properties of the nixtamalization products and by-products have been fully detailed. With the aim of replacing the nixtamalization process, ohmic heating represents a promising alternative in producing high-quality flours and fewer nutrients and phytochemicals may be wasted (Ramírez-Jiménez et al. 2019; Varghese et al. 2014). In addition to this, such emerging protocol allows the reduction of water consumption and lacks wastewaters.

Nejayote, as the primary by-product of the so-called "nixtamalization" process, has been widely explored by the research community as potential source of biomolecules with health and cosmeceutical properties. By analyzing the current development works, the following concluding remarks and recommendations for the new scientist in the field are given:

- The valorization of nejayote to produce high-added-value bioproducts for food and cosmetic sectors, is identified as a current trend in the field. Herein, specific approaches, such as spray drying and membrane-based techniques have been evaluated for recovering polyphenols and AXs, among other biomolecules. While biotransformation gives the possibility of producing other high-added value molecules (e.g. vanillin and vanillyl alcohol) (Mathew and Abraham 2006). In this way, isolation of biomolecules (such as phenolics and hemicellulose) from nejayote is preferred since such chemicals can be functional in other applications in the industries. On the other hand, it is likely that most of the studies on wastewater treatment deal with the degradation of the organic matter contained in such streams, however, the developed technologies are still challenging since they are expensive and not established yet.
- Under the framework of sustainable development goals set by the United Nations, promising biotechnological approaches dealing with the production of renewable energies (such as bio-methane and hydrogen) have been successfully developed. At this point, it is likely that nejayote will be continuously exploited for its potentiality in fermentations as a raw substrate.

Disclosure statement

The authors declare there is no conflict of interest.

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