Ion Exchange Filtration
Handling/Processing

Identification of Petitioned Substance

<table>
<thead>
<tr>
<th>Chemical Names:</th>
<th>Lewatit</th>
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<tbody>
<tr>
<td></td>
<td>Amberlite™</td>
</tr>
<tr>
<td></td>
<td>POROST™</td>
</tr>
<tr>
<td></td>
<td>Amberlyst™</td>
</tr>
<tr>
<td>Other Names:</td>
<td>CAS Numbers:</td>
</tr>
<tr>
<td>Ion exchange resin</td>
<td>7440-44-0 (activated carbon)</td>
</tr>
<tr>
<td>Ion exchange polymer</td>
<td>Variable with each ion exchange polymer</td>
</tr>
<tr>
<td>Ion exchange beads</td>
<td></td>
</tr>
<tr>
<td>Polystyrene</td>
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<tr>
<td>Anionic exchange resin</td>
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<tr>
<td>Cationic exchange resin</td>
<td></td>
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<tr>
<td>Strongly acidic resin</td>
<td></td>
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<td>Weakly acidic resin</td>
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<tr>
<td>Strongly basic resin</td>
<td></td>
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<tr>
<td>Weakly basic resin</td>
<td></td>
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<tr>
<td>Zeolite</td>
<td></td>
</tr>
<tr>
<td>Activated carbon</td>
<td>EC No. 231-153-3 (activated carbon)</td>
</tr>
<tr>
<td>Clay</td>
<td>RTECS No. FF5250100 (activated carbon)</td>
</tr>
<tr>
<td>Clay Minerals</td>
<td>Variable with each ion exchange polymer</td>
</tr>
<tr>
<td>Trade Names:</td>
<td></td>
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<tr>
<td>Dowex™</td>
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</table>

Summary of Petitioned Use

Ion exchange filtration is a process through which the composition of treated foodstuffs and water sources used in food processing are purified by removing undesired substances. However, unlike a typical filtration process, where substances are simply removed by some structural characteristic (e.g., size, solubility), ion exchange filtration uses an exchange of substances. These exchanges are often based on acid–base chemistry, where one acidic or basic group in the treated substance is exchanged for a different acid or base from the filtration material. While acid and base functionalities are common in ion exchange filtration materials, the process may also be governed by electrostatic attractions. For example, a cation (positively charged ion) in the substance is exchanged for a cation from the filtration material. In all cases, the filtration process results in the exchange of one chemical species from the substance with another from the filtration material with a similar charge (+ with +, - with -) to maintain net neutrality.

Ion exchange filtration is a purification technique that is used in many handling and processing applications across many industries, including food, water treatment, pharmaceuticals, and chemical and biochemical production (Andrés et al. 1997, Skoog et al. 2007, Kammerer et al. 2011). This report was requested by the National Organic Program (NOP) for background on the technique and will focus on the uses of ion exchange filtration in the processing and handling of organic food products (USDA 2019a).

Characterization of Petitioned Substance

Composition of the Substance:

*Synthetic ion exchange materials*
Ion exchange filtration most commonly employs a synthetic, polymer-based material through which the purification takes place (Skoog et al. 2007). The filtration material can be employed through several forms, including as porous beads, adsorbent resins, or membranes (Bazinet et al. 1999, Ma and Lada 2003, González et al. 2006, Atungulu et al. 2007, Serpen et al. 2007, Skoog et al. 2007, Coca et al. 2008, Dardel and Arden 2008, Kammerer et al. 2011).

Within the polymeric materials used in ion exchange filtration, there are several formats for the material and processes through which it is used, each featuring a different composition. Acidic or cation exchange resins employ acidic functional groups (groups that give an H\(^+\)) bonded to the polymer structure. These acidic groups exchange a cation (e.g., H\(^+\), Na\(^+\), K\(^+\), Mg\(^{2+}\)) from the filtration material for another cation in the treated substance. The most common functional groups are sulfonic acids (-SO\(_3\)H) in strong acid exchange media and carboxylic acids (-COOH) in weak acid exchange media (structures shown in Table 1) (Skoog et al. 2007, Kammerer et al. 2011).

Basic or anion exchange resins employ basic functional groups (groups that give an OH\(^-\)) bonded to the polymer structure. The most common functional groups are quaternary ammonium species (positively charged nitrogen bonded to four total alkyl groups [R [-NR\(_3^+\)]]) in strongly basic ion exchange filtration materials (Table 1) (Kammerer et al. 2011). Common weakly basic functional groups include primary ammonium species (positively charged nitrogen with three hydrogens [-NH\(_2^+\)], iminium species (positively charged nitrogen including a double bond [=NR\(_2^+\), where R is an alkyl group or a hydrogen]), and nitrilium species (positively charged nitrogen with a triple bond [≡NR\(_3^+\), where R is an alkyl group or a hydrogen]) (Table 1) (Kammerer et al. 2011). In all basic media (whether strong or weak), the positively charged nitrogen bound to the exchange material is commonly associated with a hydroxide ion (OH\(^-\)), although there have also been reports of chloride (Cl\(^-\)), sulfate ions (SO\(_4^{2-}\)), and carbonate ions (González et al. 2006, Kammerer et al. 2011, Chandrasekara and Pashley 2015). The hydroxide, chloride, sulfate, or carbonate anions from the filtration material is exchanged with an anion (negatively charged ion) from the treated substance.

When the filtration material anion is hydroxide, the resin is capable of reacting with acids in the treated material to release water rather than hydroxide ions, as shown in Equation 1 below. In the case illustrated below in Equation 1, the hydroxide is initially present in the basic polymeric structure (it is electrostatically bound to the incorporated positively charged nitrogen). The presence of an initially present hydroxide as the basic anion allows two mechanisms for ion exchange: 1) The hydroxide ion (OH\(^-\)) is exchanged for another negatively charged ion (e.g., chloride [Cl\(^-\)], nitrate [NO\(_3^-\)], etc.), which provides the same mechanism for exchange as the acidic, cationic resins described above. 2) The initially present hydroxide ion (OH\(^-\)) reacts with an acidic substance in the treated food product; this is illustrated in Equation 1 below, where the acid has been simplified to a hydrogen ion (H\(^+\)), which is the active portion of acidic substances. In this second case, the remaining portion of the neutralized acidic substance will bear a negative charge and replace the hydroxide ion that was initially present in the polymer resin.

\[
\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}
\]

\text{Equation 1.}

Mixed ion exchange materials incorporate both weak acid (-COOH) and weak base (-NR\(_3^+\), =NR\(_2^+\), or ≡NR\(_3^+\)), enabling the exchange of both anions and cations simultaneously (Shimizu 1980, Chandrasekara and Pashley 2015).

Once maximum loading has been reached, the filtration media must be regenerated or recharged (discussed in greater detail in the Action of the Substance section below). Recharging solutions of...
hydrochloric and sulfuric acids or ammonium bicarbonate regenerate the initial acidic functionality (-SO$_3$H and -COOH) from the deprotonated versions (-SO$_3$- and -COO-), whose negative charge bind cationic compounds from the treated substances (Dzyazko and Belyakov 2004, González et al. 2006, Chandrasekara and Pashley 2015). The return to a neutral functionality by reaction with acids frees the cationic compounds from the filtered substances, regenerating the filtering material for further use.

Recharging solutions for basic ion exchange filtration systems are predominantly sodium hydroxide, but there are also regeneration processes that use solutions of potassium hydroxide, calcium hydroxide, ammonium carbonate, ammonium bicarbonate, and sodium chloride (González et al. 2006, Xing et al. 2007, Kammerer et al. 2011, Chandrasekara and Pashley 2015). In the regeneration of basic ion exchange materials, the anions used in the regeneration process (e.g., hydroxide [OH$^-$], chloride [Cl$^-$], carbonate [CO$_3^{2-}$]) exhibit high binding affinities with the basic functional groups present in the ion exchange material, displacing the anions removed during the purification event (Tzeng et al. 1988, González et al. 2015).

Non-synthetic ion exchange materials

Natural ion exchange substrates include zeolites, clay, and activated carbon (USDA 2002a, Skoog et al. 2007, Kammerer et al. 2011, Marakatti et al. 2014). Zeolites are crystalline structures of aluminosilicates that feature cations of alkali or alkali earth metals (metals in group 1 and 2 of the periodic table) (e.g., potassium [K$^+$], sodium [Na$^+$], calcium [Ca$^{2+}$]) that can be of either non-synthetic or synthetic origin (Kammerer et al. 2011, Marakatti et al. 2014). Activated carbon is derived from natural plant and animal sources, including bones, coals, peat, wood, and lignite (USDA 2002a, Skoog et al. 2007, Kammerer et al. 2011, Marakatti et al. 2014).

Source or Origin of the Substance:

Synthetic ion exchange materials

Ion exchange filtration uses an ion exchange polymeric material, which is a synthetic substance. The polymer can be produced in several forms, most commonly as beads, resins, or membranes (Bazinet et al. 1999, Ma and Lada 2003, González et al. 2006, Atungulu et al. 2007, Serpen et al. 2007, Skoog et al. 2007, Coca et al. 2008, Dardel and Arden 2008, Kammerer et al. 2011). Most of the polymeric substrates used in ion exchange filtration are produced with the polymerization of styrene and divinyl benzene (Figure 1) (Davankov and Tsyurupa 1990, Kammerer et al 2011, Alvarado and Chen 2014). Acidic or basic functional groups (discussed above in Composition of the Substance) are incorporated within the polymeric backbone. The function of the ion-exchange polymer dictates the identity and density of the functional groups (Davankov and Tsyurupa 1990, Skoog et al. 2007, Kammerer et al. 2011).

![styrorene and divinyl benzene](image)

Figure 1.
Non-synthetic exchange media include clay and zeolites (alkali or alkali earth compounds with aluminosilicates). Clay is naturally present in soil horizons, marine sediments, geothermal fields, volcanic deposits and weathered rock formation (USGS 1999). Zeolites are formed upon the reaction of volcanic rocks with alkaline water sources and can be found in many mineral sources (Marakatti et al. 2014). Ion exchange can also be carried out via activated carbon, which is derived from natural sources such as bones, coals, peat, wood, and lignite (USDA 2002a, Kammerer et al. 2011). However, unlike clay and zeolites, activated carbon must be manufactured rather than be used in its natural state.

**Properties of the Substance:**

Ion exchange filtration is used in a range of applications such as food processing, water purification and treatment, and pharmaceuticals (Andrés et al. 1997, Skoog et al. 2007, Kammerer et al. 2011, Marakatti et al. 2014). Given the large range of these applications, the ion exchange media used in each application are quite different and include both non-synthetic and synthetic substrates. The properties of non-synthetic substrates (clay, zeolites) and synthetic substrates (activated carbon, acidic exchange polymers, basic exchange polymers) are listed below in Table 1. Table 1 lists the most common functional groups that are associated with various types of ion exchange materials, as well as the types of ions or compounds that are typically exchanged during the ion exchange filtration process. Specifically, the exchanges with column describes the ions/molecules that are most often removed from the substance or solution being purified. These ions/molecules are removed from the treated substances and remain within the ion exchange material until they are removed through a regeneration event.

<table>
<thead>
<tr>
<th>Ion Exchange Material</th>
<th>Functional Group(s)</th>
<th>Exchanges with</th>
</tr>
</thead>
<tbody>
<tr>
<td>Activated Carbon</td>
<td>N/A</td>
<td>Gases, and compounds, depending on activation process</td>
</tr>
<tr>
<td>Clay</td>
<td>SiO$_2$, Al$_2$O$_3$, MgO</td>
<td>Cations</td>
</tr>
<tr>
<td>Zeolites</td>
<td>Aluminosilicates (AlO$_4^-$), (SiO$_4$)</td>
<td>Cations</td>
</tr>
<tr>
<td>Acid exchangers</td>
<td>Sulfonate (-SO$_3^-$)</td>
<td>Cations (M$^{m+}$)</td>
</tr>
<tr>
<td></td>
<td>Carboxylate (-COO$^-$)</td>
<td>e.g., Na$^+$, K$^+$, Li$^+$, Ca$^{2+}$, Mg$^{2+}$, Ba$^{2+}$, Pb$^{2+}$, Cd$^{2+}$, Fe$^{2+}$, Fe$^{3+}$</td>
</tr>
</tbody>
</table>
### Specific Uses of the Substance:

#### Water Purification

Ion exchange filtration is commonly used in water purification applications. The process is most commonly employed to remove undesirable tastes and smells (e.g., sulfur and sulfide \([S^2-]\) compounds, phosphate \([PO_4^{3-}]\) and other phosphorus-containing compounds) and ions that give unfavorable characteristics such as hard-water (calcium \([Ca^{2+}]\) and magnesium ions \([Mg^{2+}]\), or it removes poisonous heavy metal ions (e.g., lead \([Pb^{2+}]\), cadmium \([Cd^{2+}]\), chromium \([Cr^{6+}]\), nickel \([Ni^{2+}]\), cobalt \([Co^{3+}]\)) (Tzeng et al. 1988, Dzyazko and Belyakov 2004, Yeon et al. 2004, Chen and Wu 2005, Atungulu et al. 2007, Xing et al. 2007, Kammerer et al. 2011, WHO 2011, Alvarado and Chen 2014).

#### Food Processing

Beverage processing applications include ion exchange filtration as a means of removing undesirable substances that reduce shelf-life and promote “off-tastes” (Shaw and Buslig 1986, Andrés et al. 1997, Ma and Lada 2003, Mishra and Kar 2003, Coca et al. 2008, Kola et al. 2010). Fruit juice processing includes the removal of tartaric acid and tartrate to avoid precipitation of tartaric crystals post-processing. The incorporation of ion exchange filtration, specifically anionic exchange, results in removal of tartrate and tartaric acid, which is replaced by chloride ions (Cl\(^-\)) from the ion exchange material (Andrés et al. 1997). Fruit juice production employs ion exchange processes to remove bitter compounds and improve the taste of the final product (Shaw and Buslig 1986, Kammerer et al. 2011). Ion exchange filtration is commonly used in the production of orange, grapefruit, and other fruit juices to remove bitter phenolic compounds such as limonin (Mishra and Kar 2003, Kola et al. 2010). Ion exchange is used in beverage production to remove undesired colors and to clarify the final product (Vivekanand and Iyer 2006, Kammerer et al. 2011).

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<table>
<thead>
<tr>
<th>Basic exchangers</th>
<th>Anions (R(^-))</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="https://via.placeholder.com/150" alt="Diagram" /></td>
<td>e.g., citrate, carbonate, bicarbonate, phenolates, Organic acids</td>
</tr>
<tr>
<td><img src="https://via.placeholder.com/150" alt="Diagram" /></td>
<td>Ammonium (-NR(_4^+))</td>
</tr>
<tr>
<td><img src="https://via.placeholder.com/150" alt="Diagram" /></td>
<td>Iminium (=NR(_2^+))</td>
</tr>
<tr>
<td><img src="https://via.placeholder.com/150" alt="Diagram" /></td>
<td>Nitrilium (≡NR(^+))</td>
</tr>
</tbody>
</table>

(R = alkyl group or H)

These results have been reported across several beverage types, including wine, beer, and fruit juices (Kammerer et al. 2011).

Ion exchange filtration is also used as a clarification and decoloring step in sugar and syrup production. Sugars undergo oxidative reactions such as the Maillard reaction that result in browning or other discolorations (Serpen et al. 2007, Coca et al. 2008, Kammerer et al. 2011). Anionic exchange resins have been reported to remove discolorations and the inorganic impurities that catalyze these undesirable oxidative processes (Serpen et al. 2007, Coca et al. 2008, Kammerer et al. 2011).

Ion exchange filtration has also been applied to the removal of naturally occurring compounds within some beverage formulations. These include applications for decaffeination of coffee and tea beverages through anionic exchange resins (Dawson-Ekeland and Stringfield 1991, Kammerer et al. 2011). In addition, ion exchange polymers have also been formulated for the removal of sugar molecules to produce low-calorie beverages that are later sweetened with zero-calorie compounds (Blase and Thomas 2008, Kammerer et al. 2011).

As in water purification applications, ion exchange filtration can be used to remove heavy metal contaminants from treated food products. Ion exchange filtration has been used to successfully remove zinc, magnesium, copper, iron, and cadmium from scallop broth (Atungulu et al. 2007).

Ion exchange filtration has been investigated for its ability to isolate usable products from by-products of other manufacturing applications. These studies have explored whey and other protein isolations, purification and desalination of scallop broth, tartaric acid isolation, and pectin and phenolic compounds (Andrés et al. 1997, Bazinet et al. 1999, Schieber et al. 2003, Doultoni et al. 2004, Atungulu et al. 2007, Kammerer et al. 2011, EFSA 2018). When used in the processing of whey protein from milk and cheese manufacturing processes, ion exchange replaces calcium ions (Ca²⁺) from dairy products with sodium ions (Na⁺) from the ion exchange material to yield products that exhibit improved gelation properties (Johns and Ennis 1981, Schmidt et al. 1984).

Approved Legal Uses of the Substance:
The United States Food and Drug Administration (FDA) states that “ion-exchange resins are used in the purification of foods, including potable water, to remove undesirable ions or replace less desirable ions with one or more of the following: bicarbonate, calcium, carbonate, chloride, hydrogen, hydroxyl, magnesium, potassium, sodium, and sulfate,” at 21 CFR 173.25(b). The FDA has approved the use of ion-exchange membranes for the “processing of food,” at §173.20. The FDA has approved the use of perfluorinated ion exchange membranes for “use in the treatment of bulk quantities of liquid food,” at §173.21.

The FDA has approved ion exchange filtration materials for a wide range of uses in the processing and treatment of food products and water sources. These applications include the use of ion exchange filtration “to minimize the concentration of naturally occurring trace minerals,” in the production of sugar beet extract at 21 CFR 172.585. The FDA has listed “anionic ion-exchange resins permitted by §173.25 … to reduce the acidity of the food,” at §146.148. The FDA has approved the use of ion exchange resin to purify dimethyl sulfoxide used in the production of petroleum wax at 21 CFR 172.886, and “polyhydric alcohol esters of oxidatively refined (Gersthofen process) montan wax acids,” at §178.3770.

The FDA has approved the use of ion exchange purification for drug and medical applications, such as in the “sorbent cartridge” and for “sorbent regenerated dialysate system for hemodialysis,” at 21 CFR 876.5600. When used as a purification method for the production of drugs for animals, the FDA requires “a
complete description of the extraction and purification processes including the names and compositions of all solvents, precipitants, ion exchange resins, emulsifiers, and all other agents used,” when filing an application for a new animal drug, as stipulated at §514.1.

The Bureau of Alcohol, Tobacco, Firearms and Explosives (ATF) allows the use of ion exchange purification for “various application in the treatment of juice or wine,” with the stipulation that “anion, cation, and non-ionic resins, except those anionic resins in the mineral acid state, may be used in batch or continuous column processes as total or partial treatment of wine,” at 27 CFR 24.248. The ATF also allows the use of ion exchange purification in concert with nanofiltration to “reduce the level of volatile acidity in wine,” at §24.248. The ATF requires a formula to be filed for the application of ion exchange purification that results in the “removal of any volume of water from beer, filtration of beer to substantially change the color, flavor, or character,” at §25.55. The ATF allows the use of “distilled spirits used in recharging an ion exchange column,” at 27 CFR 17.152.

The EPA has approved ion exchange purification for water pretreatment at 40 CFR 421.106. The EPA has also approved ion exchange as an additional treatment option to minimize lead and copper levels in water systems at §141.83.

**Action of the Substance:**

In the literature, ion exchange filtration and adsorption filtration processes are sometimes used interchangeably, although they are different processes (Kammerer et al. 2011). Adsorption filtration removes an element, ion, or compound by nature of it “sticking” to the surface of the filtration material. Ion exchange filtration requires the replacement of bound ions (ions initially present in the filtration material) by others with the same charge and requires electroneutrality (Bazinet et al. 1999, Kammerer et al. 2011). This means that ion exchange filtration is based on the principle that if an ion is removed from the treated substance by the filtration material, it is replaced by an ion of the same charge that began in the filtration material (e.g., removal of positive ion from treated substance is replaced by a different positive ion from the filtration material). The ion exchange process is a result of electrostatic attractions between the ion of interest (ion to be removed from the treated substance) and the charged functional groups incorporated into the filtration material (Kammerer et al. 2011, Alvarado and Chen 2014). The electrostatic ion binding within the treated substance is affected by (1) the type and density of functional groups incorporated into the polymer, (2) the concentration of the ions to be removed from the treated substance, and (3) the binding affinity of the various ions (Skoog et al. 2007, Kammerer et al 2011, Alvarado and Chen 2014).

The binding affinity of ions represents the strength of their electrostatic attraction to the functional groups of the filtration material. This is affected by the charge density of the ion, where smaller and more highly charged ions (larger positive or negative charges) exhibit an increased attractive force to the filtration material (Skoog et al. 2007). When the binding affinity of the ions in the treated substance is greater than the binding affinity of the ions initially present within the ion exchange material, those ions are easily displaced, resulting in their exchange for the ions initially present within the treated substance (Skoog et al. 2007, Alvarado and Chen 2014).

Acidic exchange and cation exchange are used interchangeably in the literature. This is because exchange materials that incorporates acidic functional groups results in a negatively charged surface upon deprotonation (loss of H+ from acidic functionalities) (Skoog et al. 2007, Kammerer et al. 2011). The negatively charged functionalities (sulfonate [-SO₃⁻], carboxylate [-COO⁻]) are then able to “accept” a cation (positively charged ion [Mⁿ⁺]) in place of the lost H⁺, as depicted below in Figure 2 (Skoog et al. 2007, Kammerer et al. 2011).

Basic exchange and anionic exchange are used interchangeably in the literature. Removing basic hydroxide anions from the filtration material by neutralization by acidic groups found in the treated substance (shown in general in Equation 1) results in a positively charged surface that can “accept” an anion (negatively charged ion).
(Kammerer et al. 2011). The process shown below in Figure 2 shows that both the acidic and basic ion exchange substrates operate via a similar electrostatically driven process. Anionic exchanges for larger, less polar organic molecules (e.g., organic anions, organic acids) allow for improved efficiency once the exchange process has begun due to the increased hydrophobicity (less polar nature) of the media resulting in a greater partitioning from aqueous solutions (Kammerer et al. 2011).

A similar mechanism of action is followed when the substrate is naturally occurring clay or zeolites, although the specifics of the substrate are different. In the natural cases, the presence of the alkali or alkaline earth metals (elements in group 1 and group 2 of the periodic table, e.g., sodium, potassium, magnesium, calcium) in the initial filtration material is important in the formation of the natural crystal structure (Atkins et al. 2008, Kammerer et al. 2011). This is important since the packing of the initial crystal structure dictates the proximity of functional groups (the acidic or basic components that facilitate the ion exchange process) in space and determines the size of the channels through which the treated solution will pass (Kammerer et al. 2011).
Regeneration of ion exchange filtration materials

Ion exchange filtration requires the loss of ions initially present in the ion exchange material (transferred to the treated substance) during the exchange process to maintain electroneutrality (Bazinet et al. 1999, Kammerer et al. 2011). However, at some point the filtration material reaches its exchanged ion capacity and is no longer able to “accept” any more ions from the substance being purified (André et al. 1997, Crittenden and Thomas 1998, Zagorodni 2007, Coca et al. 2008, Kammerer et al. 2011, Alvarado and Chen 2014, Chandrasekara and Pashley 2015). This point saturation for the ion exchange filtration is known as breakthrough, since the ions/molecules to be removed from the treated substance begin to pass through the ion exchange material (Xing et al. 2007, Kammerer et al. 2011, Chandrasekara and Pashley 2015).

Chemical regeneration

Once the breakthrough point has been reached the filtration media must be regenerated or recharged. This process commonly occurs through the chemical treatment of the filtration media with a recharging solution, which varies based on the type of ion exchange that is occurring (Kammerer et al. 2011). Common commercial recharging solutions include water, hydrochloric acid (HCl), sulfuric acid (H₂SO₄), ammonium bicarbonate (NH₄HCO₃), ammonium carbonate ((NH₄)₂CO₃), sodium hydroxide (NaOH), potassium hydroxide (KOH), calcium hydroxide (Ca(OH)₂), sodium chloride (NaCl), potassium chloride (KCl), and ammonium chloride (NH₄Cl) (Crittenden and Thomas 1998, Dzyazko and Belyakov 2004, González et al. 2006, Xing et al. 2007, Zagorodni 2007, Coca et al. 2008, Kammerer et al. 2011, Chandrasekara and Pashley 2015).

Thermal regeneration

Thermal regeneration processes use changes in temperature to influence the binding affinity of ions once breakthrough has been reached (Chandrasekara and Pashley 2015). As temperatures are increased the electrostatic attractions between the ion exchange material functional groups and the ions collected from the treated substance are weakened (Bolto et al. 1968, Chandrasekara and Pashley 2015). The changes in binding affinity can be significant, with reports of an eight-fold reduction in ion-filtration material interactions when temperatures are increased from 30 °C (slightly above room temperature) to 80 °C (Chanda et al. 2009). The reduced binding affinity of ions at higher temperatures allows for reduced concentrations and amounts of chemical treatments required to complete the regeneration process, and also the use of less reactive recharging solutions (e.g., sodium chloride vs. sodium hydroxide) (Chanda et al. 2009, Chandrasekara and Pashley 2015).

The ability of thermal regeneration, and the relatively low reactivity profiles of the required recharging solutions has brought reports of recyclable recharging chemicals. These include the use of ammonium bicarbonate (NH₄HCO₃), which incorporates acidic (ammonium, [NH₄⁺]) and basic (bicarbonate, [HCO₃⁻]) ions that is able to recharge acidic and basic ion exchange materials (Ng et al. 2006, Chandrasekara and Pashley 2015). The ammonium cation is able to displace the cations that have been collected from the treated substance. The ammonium ion is also able to react with acidic ion exchange materials, where it acts as an acid by donating H⁺ to reform the neutral acidic functional groups, as shown in Equation 2 below. The resulting product is ammonia (NH₃), which is a gas at room temperature, enabling its removal from the ion exchange material during the regeneration process (Chandrasekara and Pashley 2015).
The basic bicarbonate anion is able to displace the anions that have been collected from the treated substance. Once the bicarbonate anion is electrostatically bound by the ion exchange material, it can undergo a reaction to produce a hydroxide ion (OH⁻), regenerating the ion exchange material (Chandrasekara and Pashley 2015). However, unlike the acidic ammonium, the bicarbonate ion reacts with water from the solution rather than the functional groups on the ion exchange material, as shown below in Equation 3. The hydroxide ion (OH⁻) produced remains with the positively charged functional groups present within basic ion exchange materials, completing the regeneration process (Chandrasekara and Pashley 2015).

\[ \text{HCO}_3^- + \text{H}_2\text{O} \rightleftharpoons \text{H}_2\text{CO}_3 + \text{OH}^- \]

Equation 3.

The carbonic acid (H₂CO₃) that is produced from the interaction of bicarbonate and water is unstable at the elevated temperatures and spontaneously decomposes to reform water and produce carbon dioxide, as shown below in equation 4 (Silberberg 2003). Carbon dioxide (CO₂) is a gas at room temperature, which enables its removal from the ion exchange material during the regeneration process (Chandrasekara and Pashley 2015).

\[ \text{H}_2\text{CO}_3 \rightarrow \text{CO}_2 + \text{H}_2\text{O} \]

Equation 4.

The gases that result from the ammonium bicarbonate recharging solution (ammonia [NH₃, Equation 2], carbon dioxide [CO₂, Equation 4]) can be captured and recombined to reform the active ammonium bicarbonate species, as shown below in Equation 5 (Chandrasekara and Pashley 2015). The ability to capture the gaseous products of the regeneration and reform the active ammonium bicarbonate compound enables a thermal regeneration process with no net chemical use for regeneration.

\[ \text{NH}_3 + \text{CO}_2 + \text{H}_2\text{O} \rightleftharpoons \text{NH}_4\text{HCO}_3 \]

Equation 5.

Electrical regeneration processes use the application of electrical energy to overcome ion binding affinities, coupled with the electrolysis (break-down) of water to form the hydrogen (H⁺) and hydroxide (OH⁻) ions required for the chemical regeneration of ion exchange materials (Dzyazko and Belyakov 2004, Yeon et al.).
When electric voltage is applied to the ion exchange material, the ions within the material (those previously exchanged from the treated substance) begin to migrate based on their charges in relation to the applied electric field (Alvarado and Chen 2014). Specifically, positively charged ions move towards the cathode (negative charge from the applied voltage), while negatively charged ions move towards the anode (positive charge from the applied field (Xing et al. 2007, Alvarado and Chen 2014). The migration due to the applied electric field results in the accumulation of ions near the anode and cathode, and their removal from the ion exchange material.

The application of electric voltage also results in the electrolysis of water, which takes place as two distinct reactions at the anode and cathode. At the cathode (applied negative charge), water is reduced (accepts electrons) to form hydroxide ions that remain in the ion exchange material, and hydrogen, which is removed as a gas (shown below in Equation 6) (Skoog et al. 2007, Alvarado and Chen 2014). Due to the negative charge on the hydroxide ion it migrates towards the anode (applied positive charge). Over the course of the migration the hydroxide ions displace the initially bound exchange anions to regenerate the basic ion exchange material (Xing et al. 2007, Alvarado and Chen 2014).

\[
2 \text{H}_2\text{O} + 2e^- \rightarrow \text{H}_2 + 2\text{OH}^- \tag{6}
\]

In the electrolysis also includes the oxidation (loss of electrons) of water at the anode (applied positive charge), to form the hydrogen ions that react with the ion exchange material, form oxygen, which is removed as a gas (shown below in Equation 7) (Skoog et al. 2007, Alvarado and Chen 2014). Due to the positive charge on the hydrogen ion, it migrates towards the cathode (applied negative charge). Over the course of the migration, the hydrogen ions react with the negatively charged functional groups present within acidic ion exchange materials, reforming a neutral material (similar to the reaction shown in Equation 2). The neutral ion exchange material results in the loss of the electrostatic attractions that bound the cations (exchanged from the treated substance), and dramatically enhances the migration of the cations towards the cathode (Alvarado and Chen 2014).

\[
2 \text{H}_2\text{O} \rightarrow \text{O}_2 + 4\text{H}^+ + 4e^- \tag{7}
\]

Regardless of the specific type of ion exchange material, most applications use either the bed or batch process to carry out the filtration (Kammerer et al. 2011). Of the two, the bed process is the more commonly used in commercial applications due to the simpler technology required (Zagorodni 2007, Kammerer et al. 2011). In the bed process, the ion exchange material is loaded with sample until it (i.e., ion exchange beads, membrane, or resin) reaches its maximum capacity, or breakthrough. Once the material reaches breakthrough, the filtration system must be regenerated to restore its initial composition (i.e., replace H\(^+\) in acidic exchange and OH\(^-\) or anionic counter ion basic exchange for the ions removed from the treated substance) (Zagorodni 2007, Kammerer et al. 2011). The bed process may also include the incorporation of an adsorbent substance to increase the working time of the ion exchange media between regenerations (Zagorodni 2007, Kammerer et al. 2011). The most common commercially incorporated adsorbent compounds are activated charcoal (or carbon), zeolites, and silica gel. These substances also have ion exchange capabilities, which may work to remove similar compounds as the polymeric exchange material or target a broader range of ionic compounds (Kammerer et al. 2011).
Another common means for ion exchange purification is the batch process. The batch process uses several concurrent purification steps that are based on equilibrium exchange processes (Crittenden and Thomas 1998, Zagorodni 2007, Kammerer et al. 2011). In a batch process, different ion exchange or other purification materials are used to selectively remove specific ions/molecules. This enables a longer working time for each step within the overall batch process, as each step removes a smaller concentration of a specific substance rather than many substances being removed in the same step (such as what occurs in the bed process described above). The increased number of steps results in a more complicated process but also one that is more efficient because the various exchange materials can be removed and regenerated or replaced while the overall purification process simultaneously continues in the other steps (Crittenden and Thomas 1998, Zagorodni 2007, Kammerer et al. 2011). The efficiency of batch processing has resulted in its common usage in beverage and water purification applications (Kammerer et al. 2011).

Combinations of the Substance:

Due to the requirement of a regeneration process (discussed above in the Action of the Substance section) over the course of ion exchange filtrations, the most common chemical combinations with the ion exchange materials are the chemicals that are used during the regeneration event. Common commercial recharging solutions include water, hydrochloric acid (HCl), sulfuric acid (H2SO4), ammonium bicarbonate (NH4HCO3), ammonium carbonate ((NH4)2CO3), sodium hydroxide (NaOH), potassium hydroxide (KOH), calcium hydroxide (Ca(OH)2), sodium chloride (NaCl), potassium chloride (KCl), and ammonium chloride (NH4Cl) (Crittenden and Thomas 1998, Dzyazko and Belyakov 2004, González et al. 2006, Xing et al. 2007, Zagorodni 2007, Coca et al. 2008, Kammerer et al. 2011, Chandrasekara and Pashley 2015). Of the substances used in recharging solutions, hydrochloric acid, sulfuric acid, and ammonium chloride are not permitted for use in organic food production. Sodium chloride is allowed in organic production, and potassium chloride, sodium hydroxide, potassium hydroxide, and calcium hydroxide are all approved for use in organic food processing at 7 CFR 205.605. Ammonium carbonate and ammonium bicarbonate are also permitted for use in organic processing, although only as leavening ingredients, as stated at 7 CFR 205.605(b).

As described above in Action of the Substance, adsorbents are added in some applications to extend the life of the exchange media between recharging (Zagorodni 2007, Kammerer et al. 2011). Activated carbon is among the most common adsorbent substance and has been approved as a filtering aid for use in organic processing as activated charcoal at 7 CFR 205.605(b). The USDA has also approved the use of diatomaceous earth, cellulose, and perlite as filtering aids at 7 CFR 205.605.

Historic Use:

Ion exchange purification processes have been historically used for many purposes, with reports of charcoal (early activated carbon) being used by ancient Sumerians and Egyptians for purification of ores used in metal-working and bronze production (Kammerer et al. 2011). There are also early reports of ion exchange in water purification with Old Testament accounts of producing potable water from treatment with wood containing rotted cellulose, and Aristotle’s description of purifying water by treatment with sand (natural zeolites) (Kammerer et al. 2011).

More modern accounts of ion exchange began in the 1850s. Thompson (1850) and Way (1850) reported ion exchange in soils, and documented changes in organic and inorganic mineral content via natural ion exchange media (clay, zeolites). The field of ion exchange purification began to quickly develop in the 1900s. Synthetic exchange media were developed upon further study of natural sources, and by the 1930s,
they were used prominently in water softening, deionization, and purification applications (Skoog et al. 2007).

Within the realm of organic food production, ion exchange filtration processes have had a somewhat complicated and changing status. Ion exchange filtration materials are not discussed in the Organic Foods Production Act of 1990. Furthermore, ion exchange materials are not currently present in the USDA NOP regulations, found at 7 CFR 205, although activated charcoal, which has been reported as an ion exchange material derived from natural sources does appear on the National List as a filtering aid as 7 CFR 205.605(b) (Kammerer et al. 2011).

In 2010, the USDA Agricultural Marketing Service (AMS) stated that “ion exchange technology is allowed, as long as materials used are on the National List. For example: listed items sodium hydroxide, sodium chloride, not listed: hydrochloric acid,” referring to recharging solutions (USDA 2019a). On May 11, 2019, the NOP sent a memo to certifying agents stating the following:

[I]on-exchange filtration is allowed in organic processing. However, nonagricultural substances used in the ion-exchange process must be on the National List of Allowed and Prohibited Substances (National List). This includes, but is not limited to, resins, membranes, and recharging materials,

This memo was considered effective as of May 1, 2020 (USDA 2019a). However, this memo sends a contradictory message, since, as stated above, there are no ion exchange materials currently present on the National List.

On August 27, 2019, the NOP sent a memo to the National Organic Standards Board (NOSB) in response to questions by organic producers to clarify the regulations and notice of the previous May 11 memo. In the August 27 memo, the NOP requested that the NOSB provide a recommendation on the use of ion exchange filtration, clarify previous inconsistencies, and delay enforcement of the previous May 11 memo until NOP was able to review the NOSB recommendation (USDA 2019a).

**Organic Foods Production Act, USDA Final Rule:**

Ion exchange filtration is not listed in the Organic Foods Production Act of 1990 (OFPA).

Ion exchange filtration is not specifically addressed in the United States Code of Federal Regulations under the USDA organic regulations (7 CFR Part 205). The USDA organic regulations include several synthetic substances that may be used as recharging solutions in ion exchange filtration, although they are not specifically noted for that use at § 205.605(b). The USDA organic regulations do not include any synthetic resins or membranes used for ion exchange filtration at § 205.605(b), although cellulose and activated charcoal (activated carbon) are included as filtering aids at section 205.605(b). Several non-synthetic substances that may be used in ion exchange filtration can be found in the USDA organic regulations at § 205.605, although they are not specifically noted for that use, such as bentonite, diatomaceous earth, and perlite.

The USDA NOP has stated that ion exchange filtration is allowed for use in organic production to “clarify, decolor, or otherwise filter liquids using a chemical exchange process” (USDA 2019a).
International:

Canadian General Standards Board Permitted Substances List –
Ion exchange filtration is not listed in the Canadian General Standards Board Permitted Substances List (CAN/CGSB-32.311-2015).

Ion exchange filtration is not listed in the CODEX (GL 32-1999).

Ion exchange filtration is not listed in EC No. 834-151 2007 or EC No. 889/2008.

Japan Agricultural Standard (JAS) for Organic Production –
Ion exchange filtration is not listed in the JAS for Organic Production.

International Federation of Organic Agriculture Movements (IFOAM) –
Ion exchange filtration is not listed in IFOAM.

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**Evaluation Questions for Substances to be used in Organic Handling**

Evaluation Question #1: Describe the most prevalent processes used to manufacture or formulate the petitioned substance. Further, describe any chemical change that may occur during manufacture or formulation of the petitioned substance when this substance is extracted from naturally occurring plant, animal, or mineral sources (7 U.S.C. § 6502 (21)).

The most prominent ion exchange materials are synthetic polymers that incorporate specific functional groups based on their specific filtration application (discussed above in the Action of the Substance section) (González et al. 2006, Atungulu et al. 2007, Kammerer et al. 2011). The most common commercial polymer substrates are synthesized by the combination of styrene, which provides the bulk of the polymer backbone, and divinyl benzene, which is used to promote cross-linking (connections between polymer strands that provide 3D stricture), as shown in Equation 8 (Davankov and Tsyurupa 1990, Kammerer et al. 2011). The polymer structure is synthesized through a radical mechanism (movement of single, unpaired electrons) (Kammerer et al. 2011, Wade 2017). The highly reactive nature of radicals (molecules with an odd number of electrons) is stabilized due to the ability of the unpaired electron to inhabit many locations throughout the molecular structure (radical delocalization) of the aromatic substrates and their intermediates (Silberberg 2003, Skoog et al. 2007, Wade 2017). Benzoyl peroxide, shown as a catalyst in Equation 8, is a common radical initiator (source of unpaired electrons), although other initiators can be used in the polymer synthesis (Kammerer et al. 2011).
Once the backbone has been isolated (product in Equation 8), the functional groups can be introduced via traditional organic chemistry aromatic substitution reactions. Acidic functional groups, such as the sulfonate (-SO₃⁻) can be accessed through electrophilic aromatic substitutions catalyzed by strong acids (e.g., H₂SO₄), as shown below in Equation 9 (Kammerer et al. 2011, Wade 2017).

Basic functionality is installed on the polymer backbone through a two-step process. The first step is the installation of an alkyl halide group (a functional group that contains a carbon-halogen bond) onto the aromatic structure through a Friedel-Crafts alkylation reaction in the presence of a strong Lewis acid catalyst (an ion/molecule that is able to accept electrons from another molecule, e.g., AlCl₃), as shown below in Equation 10 (Silberberg 2003, Skoog et al. 2007, Kammerer et al. 2011, Wade 2017). The ammonium (group that includes a positively charged nitrogen) or other basic functionality is then installed in a second step on the middle polymer containing carbon-halogen bonds as a classic substitution reaction (a reaction in which one atom or functional group is replaced by another) (Skoog et al. 2007, Wade 2017). During the substitution reaction, the nitrogen species (NR₃ in the second step of Equation 10) acts as a nucleophile (an ion/molecule with an excess of electron density) and displaces the halogen (e.g., chlorine) as shown in Equation 10 below (Kammerer et al. 2011, Wade 2017).
Evaluation Question #2: Discuss whether the petitioned substance is formulated or manufactured by a chemical process or created by naturally occurring biological processes (7 U.S.C. § 6502 (21)). Discuss whether the petitioned substance is derived from an agricultural source.

Ion exchange filtration uses an insoluble media on which the ion exchange process takes place. This support can be a natural substance in the form of clay or zeolites derived from naturally existing minerals (Skoog et al. 2007, Kammerer et al. 2011, Marakatti et al. 2014). Activated carbon can also be used, which is derived from natural plant and animal sources such as bones, coal, peat, wood, and lignite (USDA 2002a, Kammerer et al. 2011). However, most commercial ion exchange purification processes use a synthetic polymer that bears additional functionality based on the application (Andrés et al. 1997, González et al. 2006, Atungulu et al. 2007, Kammerer et al. 2011). The polymer is synthetically produced (discussed in further detail above in Evaluation Question #1 and in Source or Origin of the Substance) in the form of beads, resins, or membranes and is neither naturally occurring, nor derived from agricultural sources.

Evaluation Question #3: If the substance is a synthetic substance, provide a list of non-synthetic or natural source(s) of the petitioned substance (7 CFR § 205.600 (b) (1)).

As stated above in Evaluation Question #2, most commercial ion exchanges processes feature a solid support in the form of a synthetic polymer (Tzeng et al. 1988, Andrés et al. 1997, González et al. 2006, Atungulu et al. 2007, Skoog et al. 2007, Kammerer et al. 2011). There are no natural sources for these polymers, as they are synthetically derived, largely from styrene and divinyl benzene monomers (shown in Figure 1 and as the reactants in Equation 8) (Davankov and Tsyurupa 1990, Kammerer et al. 2011). Both styrene and divinyl benzene are derived by the dehydrogenation (loss of hydrogen gas (H₂) that produces a carbon-carbon double bond) of ethyl and diethylbenzene, which are isolated from coal tar and petroleum sources (ATSDR 2007, Jia et al. 2019).

However, original reports of ion exchange used exchange materials that are natural or derived from natural sources in the form of clay, zeolites, and activated carbon/charcoal (Skoog et al. 2007, Kammerer et al. 2011, Marakatti et al. 2014). Of these three alternatives to polymeric ion exchange substrates, both clay and zeolites are both nonsynthetic and naturally occurring, with their sources being described in greater detail in the Source or Origin of the Substance section above (USGS 1999, Skoog et al. 2007, Kammerer et al. 2011, Marakatti et al. 2014).

Evaluation Question #4: Specify whether the petitioned substance is categorized as generally recognized as safe (GRAS) when used according to FDA’s good manufacturing practices (7 CFR §205.600 (b)(5)). If not categorized as GRAS, describe the regulatory status.

The FDA has not granted GRAS status to any ion exchange material. The FDA has classified several ion exchange specific resins as a food contact surfaces (FDA 2000a, FDA 2000b, FDA 2000c, FDA 2000d, FDA 2001a, FDA 2001b, FDA 2003a, FDA 2003b, FDA 2004, FDA 2005a, FDA 2005b). The FDA has stated “ion-exchange resins are used in the purification of foods, including potable water, to remove undesirable ions or replace less desirable ions with one or more of the following: bicarbonate, calcium, carbonate, chloride, hydrogen, hydroxyl, magnesium, potassium, sodium, and sulfate,” at 21 CFR 173.25(b). The FDA has also approved the use of ion-exchange membranes for the “processing of food,” at §173.20, which has also been extended to the use of perfluorinated ion exchange membranes for “use in the treatment of bulk quantities of liquid food,” at §173.21.

Evaluation Question #5: Describe whether the primary technical function or purpose of the petitioned substance is a preservative. If so, provide a detailed description of its mechanism as a preservative (7 CFR § 205.600 (b)(4)).
The primary function of ion exchange filtration is to remove undesirable elements, ion, or compounds, desalinate, and purify food products and water sources used in food processing, not to act as a preservative. However, there have been reports that incorporation of ion exchange during processing results in a longer shelf-life, thereby the removal of some compounds provides some preservative action. These reports include vanillin, rosmarinic acid, and fruit juices (Ma and Lada 2003, Georgiev et al. 2006, Hua et al. 2007). The longer shelf-life of products that undergo ion exchange processes is attributed to the removal of compounds that produce off-flavors and/or colors, or their precursor compounds (Ma and Lada 2003). Once these compounds are removed the stability of the product is enhanced, this is especially important in commercial fruit juices, which are commonly stored and sold at room temperature (Andrés et al. 1997, Ma and Lada 2003).

Evaluation Question #6: Describe whether the petitioned substance will be used primarily to recreate or improve flavors, colors, textures, or nutritive values lost in processing (except when required by law) and how the substance recreates or improves any of these food/feed characteristics (7 CFR § 205.600 (b)(4)).

In some cases, ion exchange filtration is used to improve colors of products. This is common in the treatment of beverages and sugars and syrups, where ion exchange filtration is commonly used for clarifying and decolorizing the liquid product (Tzeng et al. 1988, Andrés et al 1997, Ma and Lada 2003, Coca et al. 2008, Kammerer et al. 2011). Clarifying (removing haziness) and decoloring are accomplished in the same manner as ion exchange filtration removes compounds that are associated with haziness or color. The process may also remove selected ions that have relatively low solubility as a means of removing precipitates (solids) that form over time (Andrés et al. 1997, Ma and Lada 2003, Kammerer et al. 2011). These applications are used to improve colors/clarity by removing compounds naturally present the treated substances. Ion exchange filtration is not used to affect changes in treated food products that are brought about by other processing.

Evaluation Question #7: Describe any effect or potential effect on the nutritional quality of the food or feed when the petitioned substance is used (7 CFR § 205.600 (b)(3)).

As described above in the Specific Uses of the Substance section, ion exchange filtration is used to remove heavy metal contaminants from water sources and food products. There are reports of a range of heavy metals removed from treated substances including iron, copper, cobalt, chromium, calcium magnesium, lead, cadmium, nickel, and zinc (Dzyazko and Belyakov 2004, Yeon et al. 2004, Chen and Wu 2005, Atungulu et al. 2007, Xing et al. 2007, Kammerer et al. 2011, WHO 2011, Alvarado and Chen 2014). Several of these metals are highly toxic (e.g., cadmium, lead), and their removal from food products will improve nutritional quality. However, other metals are important trace minerals that are essential for proper protein function (e.g., iron, magnesium, zinc) (Timberlake 2016). The full removal of essential minerals would result in the decreased nutritional of treated substances.

While mineral content may be affected by ion exchange filtration, most studies have shown that ion exchange filtration selectively removes undesired compounds, while having no significant effect on the levels of proteins and amino acids (Atungulu et al. 2007, Serpen et al. 2007, Coca et al. 2008). Therefore, significant changes to larger molecules (e.g., amino acids, protein, carbohydrates) are unlikely to be affected by ion exchange treatments.

Some ion exchange processes have been selectively developed to alter nutritional values of food products by removing sugars to create low-calorie or diet products from sources that contain high sugar content.
(e.g., fruit juices) (Blase and Thomas 2008, Kammerer et al. 2011). Similarly, ion exchange filtration has been used to isolate proteins from by-products of other food processes, which has been used in the production of whey, pectin, and tofu (Schmidt et al. 1984, Bazinet et al. 1999, Schieber et al. 2003, Doulthani et al. 2004, Kammerer et al. 2011, EFSA 2018).

**Evaluation Question #8:** List any reported residues of heavy metals or other contaminants in excess of FDA tolerances that are present or have been reported in the petitioned substance (7 CFR § 205.600 (b)(5)).

At the time of publication of this report, the author found no published reports of ion exchange filtration contributing to heavy metals or other contaminants. Ion exchange filtration is often used as a purification method to remove heavy metals and contaminants from food and water sources (Andrés et al. 1997, Skoog et al. 2007, Kammerer et al. 2011, Marakatti et al. 2014).

**Evaluation Question #9:** Discuss and summarize findings on whether the manufacture and use of the petitioned substance may be harmful to the environment or biodiversity (7 U.S.C. § 6517 (c) (1) (A) (i) and 7 U.S.C. § 6517 (c) (2) (A) (i)).


The most likely cause for harm to the environment and/or biodiversity is by improper handling of recharging solutions. As discussed above in the regeneration of ion exchange filtration materials subsection of the Action of the Substance section, there are several methods that are used for the regeneration of ion exchange materials. The specific type of regeneration process is dependent on the specific ion exchange material, its functional groups, and the substance that is being treated (Dzyazko and Belyakov 2004). The composition of the filtration material, and the ions that need to be removed in the regeneration event, make some materials incompatible with the relatively unreactive recharging solutions frequently used in thermally and electrically driven regeneration processes (Dzyazko and Belyakov 2004, Alvarado and Chen 2014, Chandrasekara and Pashley 2015).

When the ion exchange material is regenerated through only chemical regeneration processes relatively high concentrations (~1 M (molar)) of strong acids (hydrochloric and sulfuric acids) and strong bases (sodium and potassium hydroxide) are commonly used (González et al. 2006, Xing et al. 2007, Alvarado et al. 2014, Chandrasekara and Pashley 2015). The recharging solutions are often highly acidic or basic (depending on the type of ion exchange media being recharged) and may also contain contaminants or heavy metals that were collected by the ion exchange material (Andrés et al. 1997, González et al. 2006). These concentrated and reactive solutions may not be properly handled, and there are reports that the recharging solutions are often not properly neutralized (treated with either acid or base to bring them to a neutral pH) (González et al. 2006).

Regardless of the regeneration process, each one will result in solutions containing the ions that were removed from the ion exchange material during the regeneration process. Given the wide range of applications and substances treated through ion exchange filtration it is impossible to discuss the resulting solutions in general terms. However, the resulting regeneration solutions may contain heavy metals,
organic acids, and concentrated salt solutions. Proper treatment of these potentially reactive and environmentally harmful mixtures is essential for protection of the environment and biodiversity.

Many synthetic polymers are derived from petroleum deposits and their by-products (Lligadas et al. 2013, Zhu et al. 2016). Common ion exchange resins fall into this petroleum category (described above in Evaluation Question #3), with components primarily derived from alkyl benzenes found in coal tar and petroleum (ATSDR 2007, Lligadas et al. 2013, Zhu et al. 2016, Jia et al. 2019). Given the sources of these substances, the production of some ion exchange resins is connected to the environmental impacts and risks associated with the isolation of petroleum feedstocks as well as the synthetic isolation and production of these chemical commodities. However, there are many types and sources of polymers and ion exchange resins, not all of which are isolated from petroleum. There are many alternative polymers that are produced from renewable biomass, such as terpenes (from trees), vegetable oils, and starches and carbohydrates (from corn, etc.) (Lligadas et al. 2013, Zhu et al. 2016). The use of these renewably sourced polymers within ion exchange filtration processes has not been well reported at the time of this publication.

Evaluation Question #10: Describe and summarize any reported effects upon human health from use of the petitioned substance (7 U.S.C. § 6517 (c) (1) (A) (i), 7 U.S.C. § 6517 (c) (2) (A) (i)) and 7 U.S.C. § 6518 (m) (4)).

There have been few reports on the effect of ion exchange filtration on human health. Most of the reports investigate the effects of ion exchange in water purification applications (WHO 2011). These reports look at the water softening process, in which calcium (Ca²⁺) and magnesium ions (Mg²⁺) that are responsible for hard water scale and soap scum are replaced with sodium ions (Na⁺) (WHO 2011). The focus of these reports are the contributions of hard water to dietary intake of essential calcium and magnesium, of which the WHO has stated that hard water may contribute to daily essential mineral intake, especially for people that have limited intake through their diet (WHO 2011). In connection to the calcium and magnesium content of hard water, there have been some reports that hard water is linked with improved cardiovascular health (Pocock et al. 1981, Rubenowitz et al. 1999). However, the WHO has stated that more research needs to be conducted to establish whether the findings are indeed correlated to the mineral content of hard water sources (WHO 2011).

Studies have shown that ion exchange resins degrade over time with some regeneration methods that employ a combination of chemical and thermal methods being the most problematic (Zagorodni et al. 2002, Arm et al. 2005, Chambree et al. 2005, Singare et al. 2011). The deterioration of resin material results in the loss of resin activity in terms of efficiency or capacity (Arm et al. 2005, Chambree et al. 2005, Singare et al. 2011). In one method of degradation, the polymeric structure remains intact although the number of ion exchange sites is reduced. This reduction is due to strong adsorption of compounds from purified food products or additional crosslinking (bonding) between existing functional groups on the polymer or with adsorbed materials (Zagorodni et al. 2002). Additionally, the ion exchange material may lose functional groups that were initially present (Zagorodni et al. 2002, Arm et al. 2005, Chambree et al. 2005, Singare et al. 2011). The loss of functional groups is thought to occur primarily during the regeneration process (Zagorodni et al. 2002, Arm et al. 2005, Chambree et al. 2005, Singare et al. 2011). These studies on the degradation of ion exchange resins do not comment on potential health effects from the resin material leaching into treated food products. At the time of this report, no published studies on human health effects of ion exchange degradation were found.

Evaluation Question #11: Describe any alternative practices that would make the use of the petitioned substance unnecessary (7 U.S.C. § 6518 (m) (6)).
There are several practices that may be used in place of ion exchange filtration to achieve specific aspects required in food processing. In terms of clarifying and decoloring food products, alternatives include chemical treatments (precipitation), centrifugation, and temperature treatments (USDA 2016a). Centrifugation would result in the separation of suspended solid particles, accumulating them through density differences and enable purification through mechanical filtration (Garrett and Grisham 2013).

Chemical treatments have been historically used to remove specific components of food products due to their reactivity. These include the neutralization of acids and bases and the precipitation (forming of a solid) by adding another soluble ionic compound (Andrés et al. 1997, Timberlake 2016, USDA 2016a). In the case of acid neutralization, sodium hydroxide, potassium hydroxide, calcium hydroxide, calcium carbonate, sodium carbonate, sodium bicarbonate, and potassium carbonate are basic substances that appear on the National List at 7 CFR 205.605 (USDA 1995a, USDA 1995b, ODHS 1998, NHMRC 2011, USDA 2016b, USDA 2018). For base neutralization ascorbic, citric, lactic, and tartaric acids are traditional pH regulators in food products and appear on the National List at 7 CFR 205.605 (USDA 2011, USDA 2015a, USDA 2015b, USDA 2019b). The neutralization of acidic and basic compounds in the treated substance may result in significant changes to the solubility of the neutralized compound, enabling it to be removed via traditional filtration techniques (Timberlake 2016, Wade 2017).

However, chemical changes may also result in unintended reactions within the complex mixtures present in most food products, which may result in unintended changes to flavors and colors. Specifically, carbonate anions (CO₃²⁻) and tartaric acid have relatively low solubility, and chemical treatments with them may result in enhanced haziness of the treated substance (carbonates and organic acids are a target ion for removal by ion exchange filtration).

Another alternative to ion exchange filtration is to use temperature fluctuations to take advantage of changes in the solubility profiles of compounds. As temperatures are decreased, compounds exhibit reduced solubility, which may allow for the removal of selected compounds (e.g., organic acids) through mechanical filtration (Silberberg 2003, Timberlake 2016).

Given the wide range of applications and substances treated through ion exchange filtration it is impossible to discuss the resulting solutions in general terms. In some cases, the alternative treatments discussed above may promote the neutralization, precipitation, and/or removal of compounds within the treated substance. However, in other cases these chemical agents or temperature changes may be incompatible with the treated substance or may result in undesired reactions that impact the taste, color, texture, or nutritional qualities of the substance.

Evaluation Question #12: Describe all natural (non-synthetic) substances or products which may be used in place of a petitioned substance (7 U.S.C. § 6517 (c) (1) (A) (ii)). Provide a list of allowed substances that may be used in place of the petitioned substance (7 U.S.C. § 6518 (m) (6)).

While commercial ion exchange filtration is most affected using synthetic polymers bearing specific functional groups, there are also natural materials that can be used to facilitate ion exchange. Clay and zeolites can be found in natural sources with mineral content that facilitates ion exchange (USDA 2002a, Skoog et al. 2007, Kammerer et al. 2011, Marakatti et al. 2014). The relative effectiveness of these natural sources of ion exchange materials compared to their synthetically derived polymeric counterparts is dependent on the specific application, amount, and substance that is being treated. There are no modern reports that compare these materials with the synthetic ion exchange materials. However, given nearly all commercial ion exchange processes use synthetic materials, it can be assumed that the polymeric materials are more effective, more efficient, or more cost-effective (Bazinet et al. 1999, Ma and Lada 2003, González et
The USDA has approved the use of several synthetic filtering aids. Activated charcoal can be sourced from natural materials and offers an additional substrate capable of ion exchange filtration and has been approved for organic use at 7 CFR 205.605(b) (USDA 2002a). The USDA has approved cellulose as a synthetic filtering aid at 7 CFR 205.605(b). Cellulose enables purification by filtration but works through absorption of elements, ions, and molecules, rather than via ion exchange (USDA 2016a). The ability of cellulose to absorb liquids makes it an efficient filtering aid (USDA 2016a). Cellulose is a component of many plants, and therefore, it has many sources. However, the plant material must undergo significant processing to remove lignin and other plant materials and to ensure the purity of the final product (USDA 2016a).

The USDA has approved the use of non-synthetic filtering aids. Diatomaceous earth as a filtering aid at 7 CFR 205.605(a). Diatomaceous earth is powdered or crumbled sedimentary rock that is known for its high porosity (USDA 1995c). The porous nature of the substance allows for its utility in filtration processes. The USDA has approved perlite as a filtering aid at 7 CFR 205.605(a). Perlite is a dense volcanic rock containing aluminosilicates, which can be crushed into a powder for use as a filtering aid, and alternative to diatomaceous earth (USDA 1996).

Once again, the wide range of applications and substances treated through ion exchange filtration make it impossible to discuss non-synthetic alternatives in general terms. In some cases, the non-synthetic alternative treatments discussed above may an effective alternative to synthetic ion exchange filtration processes. However, in other cases these filtration techniques and materials, may be unable to remove specific compounds, or may remove undesired compounds from the treated substance.

Evaluation Information #13: Provide a list of organic agricultural products that could be alternatives for the petitioned substance (7 CFR § 205.600 (b) (1)).

There are no organic agricultural products that serve as an adequate alternative to ion exchange filtration for the purification, clarification, decoloring, and desalination applications that are used throughout food processing.

Report Authorship

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