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Guayule (*Parthenium argentatum*) resin: A review of chemistry, extraction techniques, and applications

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ABSTRACT

Guayule (*Parthenium argentatum* Gray) is a perennial shrub of the Asteraceae family native to the arid southwestern U.S. and northern Mexico that produces high-quality, hypoallergenic natural rubber. Guayule processing produces three output streams: high-molecular weight rubber as the primary product, a mixture of resin/low-molecular weight rubber, and a fine, dry woody bagasse. The goal of this review was to evaluate guayule resin as a source of natural resin and secondary metabolites in order to overcome obstacles to commercialization by maximizing the total economic value of extractable fractions, increasing the scale and robustness of extraction methods, while concurrently lowering the operational costs and environmental impacts of separations. Based on the literature and current market prices, the guayule resin applications with the highest economic potential are residential pesticides (with a specific price range of 1.1–6.8 USD/kg), paints and coatings (0.7–6.0 USD/kg), wood preservative coatings (0.8–6.0 USD/kg), amine-epoxy strippable coatings (0.8–2.5 USD/kg), and adhesives (0.125–1.60 USD/kg). The separation methods with the greatest potential for scale-up are pressurized solvent extractions, such as supercritical CO_2 extraction, due to their reduced extraction times, energy requirements, and solvent consumption.

1. Introduction

Guayule is a perennial shrub of the Asteraceae family (over 20,000 species) native to the arid region of the southwestern U.S. and north-central Mexico. Guayule is a source of high-quality natural rubber (*cis*-1,4-polyisoprene), mostly identical to the rubber from the *Hevea brasiliensis* tree, which supplies 90 % of the world's natural rubber for applications in the tire, medical, pharmaceutical, and food industries (Mooibroek and Cornish, 2000). Annual global consumption of natural rubber is more than 12 megatons; by 2030, consumption is predicted to rise to 30 megatons (Cornish, 2017). Natural latex rubber from *Hevea* trees contains the protein responsible for type 1 latex allergies (Ownby et al., 1996). Guayule rubber does not contain the allergenic proteins, providing the potential for hypoallergenic latex materials (Cornish, 1996). Guayule also does not contain laticifers as *H. brasiliensis* does; the rubber is stored in the cytosol of parenchyma tissues (Backhaus and Walsh, 1983) and mesophyll cells of leaves (Madhavan and Benedict, 1984), and thus requires different methods for rubber extraction. An acre of two-year-old guayule plants will typically yield 0.5–1 Mg of rubber (Cornish et al., 2006). Guayule plants can tolerate some intense growing conditions: temperatures ranging from −18 °C to +49 °C (van Beilen and Poirier, 2007), rainfall as low as 350−800 mm/year, a variety of soils, and pest pressures (Foster and Coffelt, 2005).

Despite all of these advantages, commercialization of guayule rubber has been slow. Among the reasons for poor commercialization of guayule are the low cost of *Hevea* rubber (approximately 1.30–2.50 USD/kg in 2020); low rubber yield (around 5–7 % by weight for a 3- to 5-year-old shrub); significant costs of cultivation, harvesting, and processing; and no efficient value-added use of the co-products (resin and bagasse). According to a recent techno-economic analysis of guayule rubber production (Sproul et al., 2020), a baseline minimum selling price of 3.08 USD/kg of guayule rubber was reported, with resin and bagasse being sold at 1.00 USD/kg and 0.10 USD/kg, respectively. The three parameters that largely impact rubber production cost are harvest yield, biomass rubber content, and resin value; improving the resin value can reduce the minimum selling price of guayule rubber as much as 50 %

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(Sproul et al., 2020). The feasibility of guayule in the Mediterranean region was investigated by Sfeir et al. (2014) and Snoeck et al. (2015).

The first record of guayule rubber use was in the early 1500s (pre-European contact) by native peoples of Mexico for making balls for use in games (Lloyd, 1911). Guayule was first used for commercial production of rubber in the late 1800s (Bonner, 1991; Singh, 2010). Since then, there have been four general periods of guayule commercialization efforts in North America, summarized in Fig. 1.

Guayule rubber can be extracted from the dried shrub using solvent extraction, or from freshly-harvested shrub using aqueous (latex) extraction (Cornish, 1996; Cornish and Backhaus, 1990; Jones, 1948; Schloman et al., 1996). A resinous liquid (a mixture of low-molecular-weight rubber and secondary metabolites) and bagasse are the residues from guayule rubber extraction (Wagner and Schloman, 1991). The physical properties of natural rubber are strongly influenced by the non-rubber constituents of the guayule plant (Monadjemi et al., 2016). As the non-rubber components, especially lipids (fatty acids like linoleic and linolenic, and triglycerides), facilitate the degradation of rubber (mostly by oxidation of polymer chain) (Keller et al., 1981), the separation of resin is a crucial step for producing high-quality rubber from guayule. A high content of resin in extracted dry rubber lowers the bulk viscosity of the final rubber product, so only a selective extraction and deresination process results in a rubber product similar to *Hevea* rubber (Schloman, 2005).

A substantial amount of resin (generally more than the amount of rubber) is biosynthesized in guayule (Abdel-Haleem et al., 2018), about 6–15% of the dry weight of the plant (Schloman, 1988a, 1988b). Both rubber and resin are produced in the epithelial cells of resin ducts (Joseph et al., 1988). Resin is accumulated in schizogenous canals and secreted into the duct lumen in leaves, stems, and roots (Gilliland et al., 1988; Joseph et al., 1988). Resin yield from acetone extraction varies significantly from 5.5−11 wt.% (Sidhu et al., 1995) based on cultivation site, harvest date, shrub strain, shrub age, and processing procedures (Schloman et al., 1986). Resin concentration has been reported at 5.5–7 dry wt.% for 2-year-old plants, and 6–8.5 dry wt.% for 3- and 4-year-old plants, but there is no correlation between total biomass yield and resin concentration (Estilai, 1991; Sidhu et al., 1995).

Unlike other natural resins, which are extracted as the main product and are the primary source of revenue (e.g. balsam and pine), guayule resin currently contributes little to the economic feasibility of guayule as an industrial crop. The objective of this review is to define efficient separation and characterization methods for natural resin compounds to guide the development of applications for guayule resin at commercial scale. A secondary objective of this review is to evaluate the economic and environmental case for large-scale natural rubber and resin extraction.

2. Guayule resin composition

Guayule resin contains a wide variety of secondary metabolites (sesquiterpene esters, triterpene alcohols, fatty acids) and is soluble in polar solvents such as alcohols, esters, and ketones. Guayule resin composition varies with shrub strain, harvest date, cultivation site, and processing history (Schloman et al., 1986). Among the limited studies on guayule resin, inconsistency between results has been linked to differences in extraction and pretreatment methods, on top of factors like genotype, plant age at the time of harvest, and cultural practices (Cheng et al., 2020). These factors have been shown to affect levels of sesquiterpene esters and fatty acid triglycerides (Schloman et al., 1983). Around 50 different classes of chemicals have been identified in guayule resin (Banigan et al., 1982). These are often divided into three main groups: terpenes/terpenoids, fatty acids, and low-molecular-weight rubber (LMWR).

2.1. Essential oils

The major groups of volatile fractions in the essential oil extracted from fresh guayule biomass include terpenes (70–83 %), oxygenated terpenes (6–10 %), sesquiterpenes (8–14 %), and sesquiterpene alcohols (3–6 %); α- pinene is the most dominant compound (Haagen-Smit and Siu, 1944; Scora and Kumamoto, 1979). These volatile fractions are produced more rapidly than rubber during periods of guayule vegetative growth (Schloman et al., 1986). These volatile compounds are synthesized via methylerythritol phosphate (MEP) and mevalonic acid pathways (Fig. 2). Geranyldiphosphate (GPP), the precursor for all monoterpenes (C_{10}), comes from the combination of dimethylallyl diphosphate (DMAPP) and isopentenyl diphosphate (IPP).

2.2. Sesquiterpene esters

The major sesquiterpene compounds, representing 10−15 wt.% of the resin, are guayulins (Sidhu et al., 1995; Watkins et al., 1985). Guayulins are both isoprenoids and aromatic acid esters (Teetor et al., 2009). Guayulins A and B are sesquiterpene esters of *trans*-cinnamic and *p*-anisic acids, respectively. A whole guayule resin contains approximately 8−14 wt.% of guayulin A and 0.5−3 wt.% of guayulin B, mostly concentrated in the woody stems (Schloman et al., 1983; Sidhu et al.,

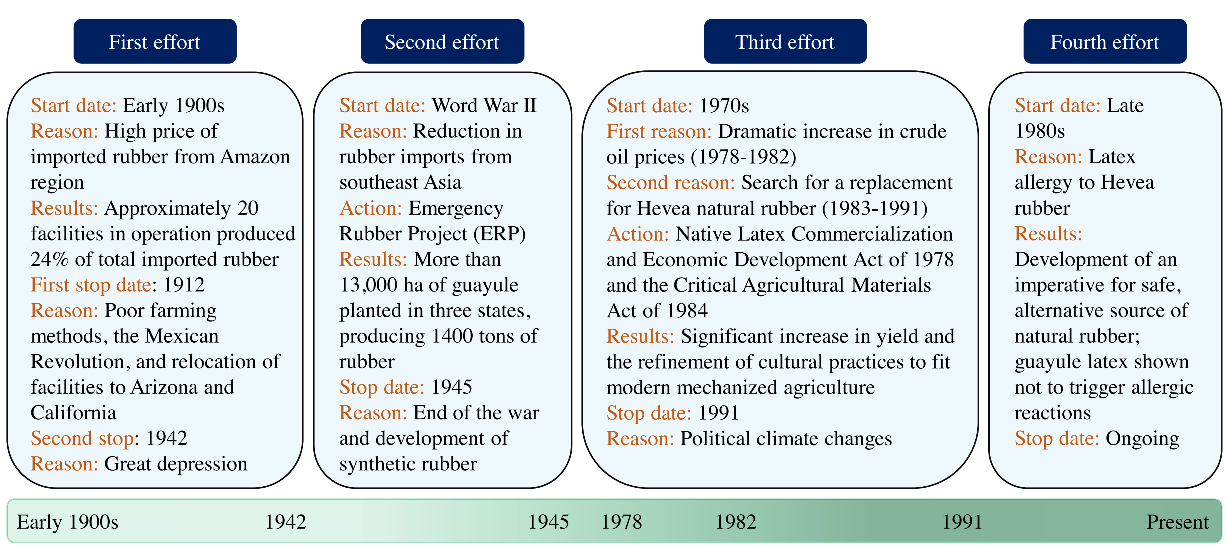


Fig. 1. Four periods of commercialization effort for guayule rubber. Elaborated from Bonner (1991); Huang (1991); Wright et al. (1991); Ray (1993); Foster and Coffelt (2005); Ownby et al. (1996); Siler et al. (1996); Cole et al. (1991); Finlay (2009), and Jones (1948).

1995; Teetor et al., 2009). The concentration of guayulin A is highly dependent on season (Schloman et al., 1986). Guayulins C and D are likely formed spontaneously through oxidation/degradation pathways of guayulins A and B, and constitute 0.1−3 wt.% and 0.1−1 wt.% of the resin, respectively (Rozalén et al., 2021; Schloman et al., 1983). The highest concentrations of guayulin C (1400−1650 mg/kg) and guayulin D (330−370 mg/kg) are found in the leaves (Spano et al., 2018). Rubber and guayulin A contents in stems with diameters larger than 10 mm are significantly correlated, so guayule A content could serve as a good predictor of total plant rubber content (Teetor et al., 2009).

Farnesyl diphosphate (FPP), the precursor of sesquiterpenes (C_{15}), comes from addition of two IPP molecules to DMAPP (Fig. 2). Sesquiterpenes are found naturally as hydrocarbons, or in oxygenated forms, including lactones, alcohols, acids, aldehydes, and ketones (Awouafack et al., 2013).

2.3. Triterpenoid esters

Argentatins, tetracyclic-type triterpenes, are also abundant in guayule resin, constituting 20–30 % of the total resin (Martínez et al., 1990; West et al., 1991). Argentatins A–D and iso-agentatin B are the most dominant compounds in this class. These triterpenes have shown anti-inflammatory and cytotoxic activities (Alcántara-Flores et al., 2015; Tavarez-Santamaría et al., 2020). Triterpenes (C_{30}) are derived from dimerization of FPP via mevalonic acid pathways (Fig. 2).

2.4. Fatty acids

Fatty acid triglycerides encompass 10−25 wt.% of guayule resin (Banigan and Meeks, 1953; Keller et al., 1981). Linoleic acid (C18:3) is the most abundant at 36–65 % of the total fatty acids, distantly followed by palmitic, linolenic, oleic, and stearic acids. Cinnamic acid and *p*-anisic acid were identified as two major aromatic acids in the saponifiable materials (Schloman et al., 1983). Chemical treatments before and during extraction, such as sodium hydroxide in latex extraction, can significantly change (mostly decrease) the yield of fatty acids extracted from resin (Belmares et al., 1980). Fatty acids ranging from 8 to 32 carbons have been found in some plants, synthesized by carboxylation of acetyl-CoA to form malonyl-CoA under a repeated series of reactions.

2.5. Sterols

Sterols (e.g. β-sitosterol) are another major compound class in guayule resin (Cheng et al., 2020), accounting for 9−11 wt.% (Buchanan et al., 1978; Schloman et al., 1983). Plant steroids are synthetized from 2,3-oxidosqualene ($C_{30}H_{50}O$) via the MVA pathways (Fig. 2) from the cycloartenol ($C_{30}H_{50}O$) precursors. Plant steroids are formed by a tetracyclic ring and a long chain attached at C-17.

2.6. Low-molecular-weight rubber (LMWR)

LMWR is an integral part of guayule resin. Approximately 20−40 wt.% of low-molecular-weight *cis*-1,4-polyisoprene compounds can be found in the more polar (resin) phase after extraction and precipitation of high-molecular-weight rubber (Wagner and Schloman, 1991). LMWR has a molecular weight one to two orders of magnitude smaller than those of natural rubber polymers (1×10^5-2.5×10^6 g/mol) (Angulo-Sánchez et al., 1995; Campos-López and Angulo-Sánchez, 1976). Detection and characterization of LWMR compounds is difficult, even with advanced mass spectroscopy techniques, such as high-resolution Fourier transform ion cyclotron resonance mass spectroscopy (FT-ICR MS), which can provide accurate masses of hundreds of compounds with a wide range of molecular weights and degrees of aromaticity (Cheng et al., 2020).

2.7. Water extractables

Resin may contain (depending on the extraction method) water extractables, which constituent 14–17 % of the woody tissues (Buchanan et al., 1978; Schloman et al., 1983; Traub and Slattery, 1946). On a whole-shrub basis, bagasse yielded 16 % extractables (7.5 % polyphenolics and 8.5 % polysaccharides) after sequential extraction, and 19 % extractables (6.8 % polyphenolics and 12.6 % polysaccharides) after simultaneous extraction (Schloman et al., 1988). The substantially lower level of polysaccharides in bagasse after sequential extraction results from entrainment of polysaccharides into the resin as part of the free aqueous phase.

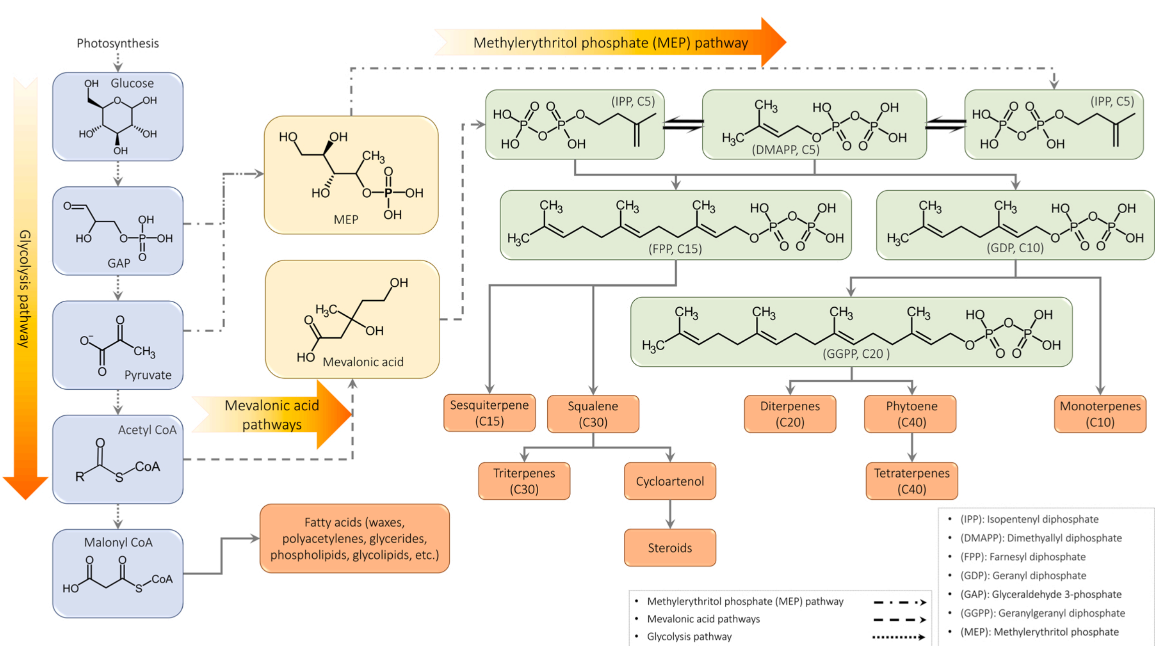


Fig. 2. Biosynthesis of terpene and fatty acid secondary compounds constituting plant resins, showing the interconnections with primary compounds and processes. Elaborated from Wink (2010); Buchanan et al. (2015); Yarnell (2007), and Springob and Kutchan (2009).

Table 1
Guayule resin composition. Compiled from Haagen-Smit and Siu (1944); Scora and Kumamoto (1979); Schloman et al. (1983); Battistel et al. (2017); Parodi (1988); Maatooq and Hoffmann (2002); Saldívar et al. (2003); de Vivar et al. (1990); Gaytán et al. (1997); Banigan and Meeks (1953); Belmares et al. (1980); Punvichai et al. (2016); Mears (1980); Shen et al. (1976); Mabry et al. (1978), and Dehghanizadeh et al. (2020).

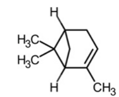
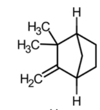
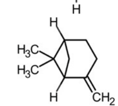
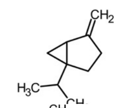
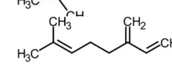
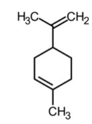
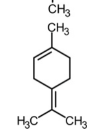
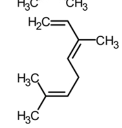
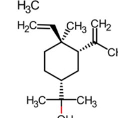
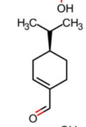
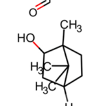
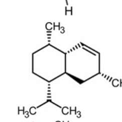
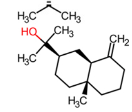
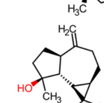
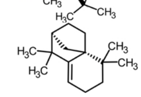
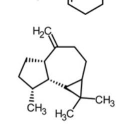
Group of compounds	Name	Chemical formula	Composition	Chemical structure
Monoterpenes	α- pinene	$C_{10}H_{16}$	0.5-3%	
	camphene	$C_{10}H_{16}$	<1%	
	β- pinene	$C_{10}H_{16}$	0.3-0.7%	
	Sabinene	$C_{10}H_{16}$	0.2-0.5%	
	β- myrcene	$C_{10}H_{16}$	~0.1%	
	limonene	$C_{10}H_{16}$	0.2-0.7%	
	terpinolene	$C_{10}H_{16}$	0.3-0.7%	
	β- ocimene	$C_{10}H_{16}$	~0.1%	
Monoterpenoids	Elemol	$C_{15}H_{26}O$	~0.2%	
	l-phellandral	$C_{10}H_{16}O$	~0.2%	
	borneol [a]	$C_{10}H_{18}O$	<0.1%	
Sesquiterpenes and sesquiterpenoids	cadinene	$C_{15}H_{26}$	0.3-0.4%	
	β-eudesmol	$C_{15}H_{26}O$	0.1-0.2%	
	spathulenol	$C_{15}H_{24}O$	–	
	isolongifolene	$C_{15}H_{24}$	–	
	allo-aromadendrene	$C_{15}H_{24}$	–	
	partheniol	$C_{15}H_{24}O$	–	

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Table 1 (*continued*)

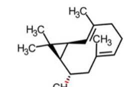
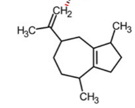
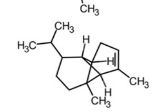
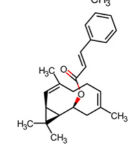
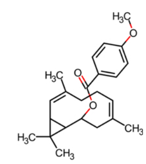
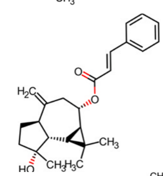
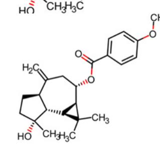
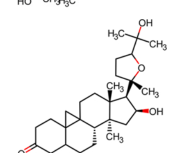
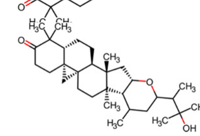
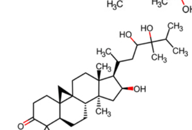
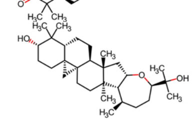
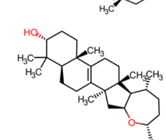
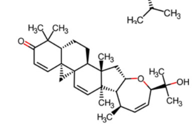
Group of compounds	Name	Chemical formula	Composition	Chemical structure
	α-guajene	$C_{15}H_{24}$	~0.1%	
	α-copaene	$C_{15}H_{24}$	–	
	α-humulene	$C_{15}H_{24}$	–	
Sesquiterpene esters	guayulin A	$C_{24}H_{30}O_2$	8-14%	
	guayulin B	$C_{23}H_{30}O_3$	0.5-3%	
	guayulin C	$C_{24}H_{30}O_3$	0.1-3%	
	guayulin D	$C_{23}H_{30}O_4$	0.1-1%	
Triterpenoids	argentatin A	$C_{30}H_{48}O_4$	4-8%	
	argentatin B	$C_{31}H_{50}O_3$	2-4%	
	argentatin C	$C_{31}H_{52}O_4$	~1%	
	argentatin D	$C_{30}H_{50}O_3$	~3%	
	argentatin E	$C_{30}H_{50}O_2$	–	
	argentatin F	$C_{30}H_{42}O_3$	–	

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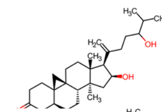
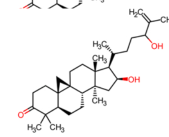
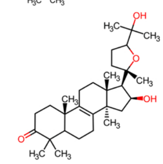
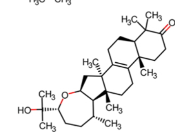
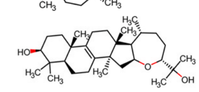
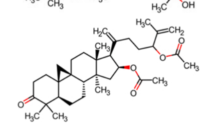
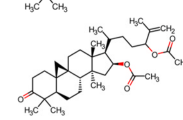
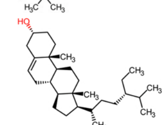
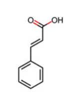
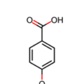
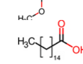
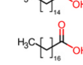
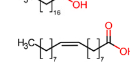
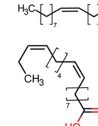
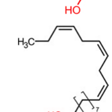
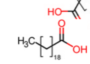
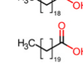
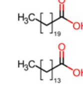
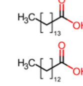
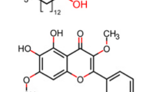
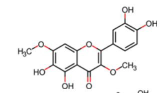
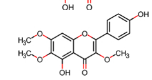
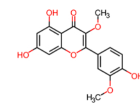
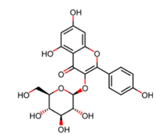
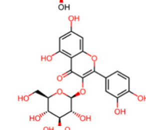
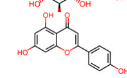
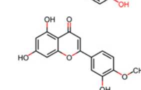
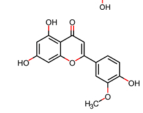
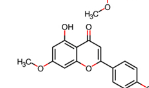
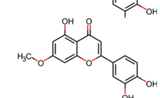
Table 1 (*continued*)

Group of compounds	Name	Chemical formula	Composition	Chemical structure
	argentatin G	$C_{30}H_{46}O_3$	–	
	argentatin H	$C_{30}H_{48}O_3$	–	
	incanilin (Iso argentatin A)	$C_{30}H_{48}O_4$	–	
	isoargentatin B	$C_{30}H_{48}O_3$	–	
	isoargentatin D	$C_{30}H_{50}O_3$	–	
	argentatin G diacetate	$C_{34}H_{50}O_5$	–	
	argentatin H diacetate	$C_{34}H_{52}O_5$	–	
Sterols	β-sitosterol	$C_{29}H_{50}O$	–	
	trans-cinnamic acid	$C_9H_8O_2$	~4%	
	p-anisic acid	$C_8H_8O_3$	0.5-1%	
	palmitic acid	$C_{16}H_{32}O_2$	1.5-2.5%	
	stearic acid	$C_{18}H_{36}O_2$	0.3-1%	
	oleic acid	$C_{18}H_{34}O_2$	1-2.5%	
Organic acids	linoleic acid	$C_{18}H_{32}O_2$	10-15%	
	linolenic	$C_{18}H_{30}O_2$	2-4%	
	eicosanoic acid	$C_{20}H_{40}O_2$	<3%	
	heneicosanoic acid	$C_{21}H_{42}O_2$	<1.5%	
	pentadecanoic acid	$C_{15}H_{30}O_2$	<4%	
	Myristic acid	$C_{14}H_{28}O_2$	<2.5%	



(continued on next page)

Table 1 (*continued*)

Group of compounds	Name	Chemical formula	Composition	Chemical structure
	6-hydroxykaempferol 3,7-dimethyl ether	$C_{17}H_{14}O_7$	–	
	quercetagetin 3,7-dimethyl ether	$C_{17}H_{14}O_8$	–	
	6-hydroxykaempferol 3,6,7-trimethyl ether	$C_{18}H_{16}O_7$	–	
	quercetin 3,3'-dimethyl ether	$C_{17}H_{14}O_7$	–	
Flavonoids	kaempferol 3-glucoside	$C_{21}H_{20}O_{11}$	–	
	quercetin 3-glucoside	$C_{21}H_{20}O_{12}$	–	
	Apigenin	$C_{15}H_{10}O_5$	–	
	Diosmetin	$C_{16}H_{12}O_6$	–	
	chrysoeriol	$C_{16}H_{12}O_6$	–	
	luteolin 7-methyl ether	$C_{16}H_{12}O_6$	–	

[a] Derived from hydrolysis of bornyl acetate.

2.8. *Other compounds*

Guayule leaves have been of recent interest as a source of phenolic-rich compounds (Piana et al., 2018; Piluzza et al., 2020). A variety of phenolic compounds and phenolic derivatives, hydroxybenzoic acids, hydroxycinnamic acids, flavanols, flavones, and anthraquinones, have been identified in the leaf biomass (Piana et al., 2018). Five phenolic compounds: neochlorogenic (5-caffeoylquinic acid), chlorogenic (3-caffeoylquinic acid), cryptochlorogenic (4-caffeoylquinic acid), isovanillic, and 3,5-DCQ (3,5-Di-O-caffeoylquinic acid) acids were recently identified in methanol extracts from guayule leaves and twigs (Piluzza et al., 2020). Mears (1980) investigated the distribution of flavonoids in the North American species of *Parthenium* and reported five major flavonoids in guayule (See Table 1).

3. Guayule resin extraction

The separation, fractionation, and purification of guayule resin are critical to any value-added use of this multicomponent mixture. Although there have been many proposed commercial applications for guayule resin fractions, few have been economically feasible due to difficulties in the separation and purification of target compounds out of the wide range of molecular weights and polarities. A few studies on separation methods have been conducted for industrial-scale applications. Fractionation of guayule resin has mostly been investigated using conventional solvent extraction techniques. Vacuum and steam distillation have been used for the separation of essential oils, with the associated thermal degradation of some non-volatile compounds (Haagen-Smit and Siu, 1944; Walter, 1944).

In latex rubber extraction, plant materials are chopped and homogenized in a alkaline aqueous medium using pebble or hammermills, followed by filtering, pressing, washing, and centrifuging to separate latex droplets; there is little extraction of resin. As result, a considerable amount of resin (~8 %) is left in the bagasse or entrained in the rubber phase (Boateng et al., 2010), lowering the initial quality of the rubber and facilitating rubber degradation/oxidation (Jones, 1948).

For dry bulk rubber extraction, resin can be obtained from three methods: flotation, sequential, and simultaneous extraction (Schloman, 2005). In flotation rubber extraction, ground guayule shrub is mixed with a caustic solution to coagulate rubber particles, then the rubber particles are deresinated using a polar solvent (acetone). This method is not suitable for the industrial scale due to the high process water requirements, some energy-intensive steps, the large volume of aqueous waste, and the low quality of produced rubber (van Beilen and Poirier, 2007; Wagner and Parma, 1989). In the sequential extraction method, resin and rubber are extracted from the ground shrub together using a polar solvent (alcohol, ketone, or ester), then rubber is isolated with a non-polar organic solvent. The two-step solvent extraction and the large amount of solvent increase operating costs and potential for solvent loss, making sequential extraction less economically feasible. In simultaneous extraction, rubber and resin are extracted using a mixture of non-polar and polar solvent, for example 20 wt.% acetone and 80 wt.% pentane, to produce a miscella (Cornish et al., 2006; Schloman, 2005); additional polar solvent is added to the miscella to precipitate rubber. Resin is

Table 2
Potential applications of guayule resin.

Category	Application	Description	References
Paints, coatings, and adhesives	Controlled-release formulation	Component of formulations that improve loading and release time of volatile compounds (linalool or carvone), and reduce the porosity of membranes	Pascual-Villalobos and López, 2013
	Resin multipolymers	Reaction of resin with unsaturated monomer for directly use in solvent-less inks, coating, and adhesives	Gumbs, 2009
	Wet-stick bio-adhesives	Bio-adhesives for bonding wet skin, such as pressure-sensitive adhesive tapes, medical tapes, wound or surgical dressings, athletic tapes, surgical drapes, or tapes/tabs for medical sensors	Gumbs, 2008
	Adhesives	Adhesives to bind different kinds of surfaces, even underwater, and to provide insect control	Nakayama, 2005
	Strippable coatings	Combination of guayule resin and epoxy polymers to make strippable coatings for the protection of equipment in storage	Nakayama, 2005
	Composite components	Combinations of wood, plastic, and guayule bagasse or resin for termite- and fungus-resistant products	Nakayama et al., 2004
	Solvent-less adhesives	Combinations of guayule resin with monomers, polymers, and initiators for solvent-less adhesives	Gumbs, 2004
	Material durability enhancers	Ingredient to improve the durability of wood/plastic composites	Chow et al., 2002
	Coatings	Impregnation of woody materials with guayule resin to provide resistance to termite attack and wood-rot	Bultman et al., 1991; Nakayama et al., 2003, 2001
	Coatings	Coatings on wood in marine and terrestrial environments to protect against crustacean borers and termites	Bultman et al., 1998
	Fire retardant	Pine wood impregnation formulation to retard ignition	Smith et al., 1996
	Anti-fouling paints	Anti-fouling paints for marine applications that are less toxic to shrimp and/or barnacles	Thames et al., 1996
	Plasticizer/adhesive	Plasticizer/adhesive modifiers in high-performance epoxy resin coatings	Thames and Kaleem, 1991
	Strippable coatings	Adhesive modifier for strippable coatings	Thames and Kaleem, 1991; Thames and Wagner, 1991
	Plasticizer for high polymers	Plasticizer for tailoring stress-strain properties	Armistead et al., 1987
	Coatings	Water and abrasion-resistant coatings	Belmares et al., 1980
Antifungal, anticancer, and antimicrobial agents	Antiplastic agent	Guayulin A and B as biological triggers for synthesizing antiplastic agent in breast cancer treatment	(Spano et al., 2018)
	Anti-cancer agent	Argentatin A and B are cytotoxic and cytostatic, respectively, for proliferating lymphocytes.	Parra-Delgado et al., 2005
	Anti-cancer	Argentatin B inhibits the proliferation of human colon cancer cell line (RKO)	Romero-Benavides et al., 2018
	Antitumor agent	Anticancer activity of argentatin A in HCT116 colon cancer cells	Tavarez-Santamaría et al., 2020
	Antitumor agent	Argentatin B inhibits the proliferation of cancer cells (HCT-5 and PC-3)	Alcántara-Flores et al., 2015
	Antifungal	Sesquiterpenoid extracts used to treat fungal infections	Maatooq and Hoffmann, 1996; Maatooq et al., 1996
	Antimicrobial	Argentatin A activity against various microorganisms	Martínez et al., 1986; Martinez Vazquez et al., 1994
	Antineoplastic agent	Guayulin A and B for synthesizing antineoplastic agent lychnostatin 1	Zoeller et al., 1994
	Anticancer agent taxol	Guayulin A and B for anticancer treatments	Zoeller et al., 1994
	Anticancer agents	Argentatin A, B, and its derivatives for cytotoxic and anti-inflammatory treatments	Parra-Delgado et al., 2005
Pesticides	Termite repellent	Natural and safe termiticide	Bajwa et al., 2017
	Bio-control agents	Argentatin A and B for growth inhibition of the fall armyworm *Spodoptera frugiperda*	Carlos et al., 2001
	Termite control agent	Sesquiterpenes and triterpenes as insect anti-feedants and toxins	Gutiérrez et al., 1999
	Cockroach pheromone	Guayulin A and B for synthesizing cockroach pheromone periplanone B	Zoeller et al., 1994
	Bio-control agents	Guayule resin negative impacts on larva stage of certain insects	Isman and Rodriguez, 1983
Rubber additives	Softener	Guayule resin as a softener in the tire sealant composition	Randall et al., 2020
	Epoxidized natural rubber	Epoxidized natural rubber from LMWR fractions to enhance physical properties of cured rubber	Schloman, 1992
	Rubber additives	Sulfurized resin to reduce hysteresis loss and increase tensile strength in conventional rubber	Schloman and Davis, 1986
	Tackifying agents	Green tackifiers and strength enhancers for improving rubber	Kay and Gutierrez, 1984, 1985
	Peptizing agent	Peptizing agent for both *Hevea* natural rubber and styrene/butadiene rubber	D.S. Winkler, 1977
Fuels	Liquid fuels	Conversion of guayule resin to liquid fuel by cracking catalysit	Gutierrez and Kay, 1983
	Biofuel feedstock	Mixture of guayule bagasse and resin	Sabaini et al., 2018
	Liquid hydrocarbons	Conversion of guayule resin to C_1-C_{10} hydrocarbons using zeolite-based catalysts	Costa et al., 1992
Miscellaneous	Asphalt binder modifier	Guayule resin as recycling agent in asphalt binders	Hemida and Abdelrahman, 2019; Lusher and Richardson, 2015
	Soil amendment	Resin-containing bagasse as soil amendment to improve lettuce growth	Schloman, 1991
	Essential oils	Essential oil from steam distillation of fresh guayule biomass for fragrance/volatiles applications	Haagen-Smit and Siu, 1944

recovered from the miscella by evaporating the solvent. The main advantage of this method is that the quality of the final rubber product, particularly the rubber viscosity and resin content, can be adjusted (Beinor and Cole, 1986). This capability is important for managing variations between guayule shrub lines and harvested biomass properties. The main disadvantages of this method are the complex separation processes for recycling solvents and removing the solvent from the rubber after precipitation (Wagner and Parma, 1988). Resin yield from sequential extraction is significantly higher (68 %) than from simultaneous extraction, however, the resin is noticeably heterogeneous and contains entrained water-soluble materials, including 1.4 wt.% ash; resin from simultaneous extraction is homogeneous and contains no water or water-soluble materials (Schloman et al., 1988). The presence of water and water-soluble materials complicate the solvent recovery process, and lower rubber yield and quality, therefore, guayule biomass is sometimes rinsed with water then dried before extraction (Black et al., 1986; Curtis, 1947). Although total guayulin, argentatin, and triglyceride contents in resin from sequential extraction are lower than those in resin from simultaneous extraction, the concentration of reactive groups is higher, increasing the potential for chemical modification with polyamines or amine-terminated polyethers (Schloman, 1988a, 1988b).

Supercritical CO_2 extraction has been investigated as an alternative for the guayule resin and rubber separation (Cornish et al., 2006; Punvichai et al., 2016). The extraction was significantly faster and better controlled than conventional liquid solvent extraction. Parameters that significantly affected the extraction yield and selectivity were pressure (10−69 MPa), temperature (60−100 °C), co-solvent ratio, and time (Cornish et al., 2006). Some (2−3 wt.%) resin remained in shrub biomass after supercritical extraction with pure CO_2 (Cornish and Marentis, 2010). Small amounts of co-solvent can significantly promote resin and rubber extraction yield; biomass particle size, moisture content, and pretreatments also play important roles in extraction efficiency. The highest yield of resin was obtained at 45−60 °C, 200−300 bar, and 60 min, using ethanol as the co-solvent (Cornish et al., 2006). Supercritical CO_2 extraction has been used for pretreating guayule bagasse to remove resin prior to its use as a feedstock for enzymatic hydrolysis (Srinivasan and Ju, 2010). A summary of these supercritical CO_2 extraction experiments is shown in Table A1.

Accelerated solvent extraction (ASE) has been investigated for guayule rubber and resin extraction at the lab scale (Cornish et al., 2013; Pearson et al., 2013; Suchat et al., 2013). Resin was extracted with a polar solvent at lower temperatures and the rubber was recovered using a non-polar solvent at higher temperatures. The maximum yield of resin and rubber (from a two-year-old shrub, oven-dried at 50 °C) was obtained after three rinses with acetone at 40 °C and three rinses with hexane at 140 °C, respectively (Pearson et al., 2013). Drying temperatures above 75 °C can degrade rubber into the acetone phase during ASE, thus overestimating resin and underestimating rubber contents (Black et al., 1983; Cornish et al., 2013; Pearson et al., 2013). Experimental results for ASE of guayule resin and rubber are summarized in Table A2.

Some other alternative techniques, including electrostatic separation, adsorptive separation, microfiltration, ultracentrifugation, and liquid cyclones have been proposed for resin extraction and purification. Clogging and high maintenance costs are the main barriers to commercialization (Thames and Wagner, 1991). The feasibility of using ultrafiltration (UF) membranes for single-step fractionation of guayule resin has been demonstrated by Jeyaseelan and Wagner (1995) and Wagner and Parma (1989). The solvents (xylene and methanol) and lower-molecular-weight fatty acids (C_{16}-C_{18}) are separable in a single-step ultrafiltration system using a ceramic membrane (Wagner and Parma, 1989). The ambient operating temperatures in ultrafiltration prevent the degradation of thermolabile constituents of guayule resin. Volatile essential oils can be separated from resin using cellulose triacetate membranes with 200 and 500 Da molecular weight cut offs, however, low flow rates and membrane fragility limit the commercial application of the technique (Daly, 1989; Jeyaseelan and Wagner, 1995).

Efficient separation and fractionation of guayule resin (or other resin-containing plants) with high chemical complexity and significant differences in compound polarity may require a combination of separation techniques. For example, using ultrafiltration alone may not be economically feasible for large-scale applications, but in combination with supercritical fluid extraction (SFE) or pressurized liquid extraction (PLE), the production cost can be significantly decreased (Sarmento et al., 2008, 2004; Spricigo et al., 2001). Selection of individual and/or combination techniques will all need to consider the associated economic tradeoffs, many of which have not yet been studied.

4. Guayule resin applications

Value-added use of guayule processing residues has the potential to reduce gross rubber production costs by 26–49 % (Schloman et al., 1986). Wright et al. (1991) suggested that research and development into such applications might even create the scenario where co-products derived from guayule resin, LMWR, and/or bagasse exceed the value of the rubber itself (Nakayama, 2005). Potential direct uses of crude guayule resin (that do not require pharmaceutical isolation and purification) include coatings, adhesives, viscosity modifiers, and plasticizers (Schloman and Wagner, 1991). Recent focus on economics has led to developments in guayule resin applications in coatings, biocontrol agents, and controlled-released formulations (Pascual-Villalobos and López, 2013). Argentatins have been reported to have antimicrobial (Martinez Vazquez et al., 1994), insecticidal (Carlos et al., 2001), and antitumoral properties (Alcántara-Flores et al., 2015). Polyphenolics, cinnamyl derivatives, and terpenoids are the most likely compounds responsible for the observed termite resistance characteristics of guayule resin (Bultman et al., 1986). The volatile fractions of guayule resin are rich in alpha-pinene, beta-pinene, and limonene—of which 30,000 tons are produced annually for fragrances and adhesives (Swift, 2004). The recently-identified phenolic compounds in the leaves and twigs have potential uses in antioxidant, anticancer, anti-inflammatory, and anti-diabetic applications (Piluzza et al., 2020). Table 2 provides a summary of guayule resin applications that have been reported but not yet commercialized.

5. Guayule resin economics

The economic feasibility of guayule rubber extraction is highly sensitive to the raw material value of the co-products (Sproul et al., 2020). The bagasse represents a relatively fixed revenue potential as it is dried biomass without an exotic compound. The resin represents a unique opportunity based on the diversity of compounds contained. The first step to estimating the resin's raw material value is to understand the range of active ingredients/products that resin can replace. Among the product replacements studied have been bio-control agents and controlled released formulations (Pascual-Villalobos and López, 2013); coatings, adhesives, viscosity modifiers, and plasticizers (Schloman and Wagner, 1991); insecticides (Gutiérrez et al., 1999); and wood preservatives (Bultman et al., 1991; Nakayama et al., 2003, 2001) (Table 2). To down-select from the long list of potential guayule resin uses, retail prices (USD/kg) and market size of key product categories were estimated using published literature and online marketplaces. Bulk retail prices, technical cost reports, and financial marketplace websites were prioritized as the most appropriate sources for wholesale product prices. When information from these sources was not available, online retail prices were used for approximations. Product categories included asphalt binders, adhesives, rubber tackifying resins, paints and coatings, soil amendments, and essential oils (Tables A3 and A4). Using this data, a landscape of potential guayule resin product prices was created (Fig. 3).

The distribution in Fig. 3 shows the resin can be used in high- and low-price products. In general, the low-price products have the largest market sizes. Conversely, products with the highest retail price are typically for smaller market sizes: antifouling paints, essential oils, marine wood coatings, wood preservative coatings, and pesticides. "Paints and Coatings" was added to account for the wider variety of coating types and paints with resin applications. Asphalt binders, soil amendments, and rubber tackifying resin are lower in price, while solvent-based, solventless, and medical spray adhesives have intermediate prices. The wide range of specific prices is attributed to variation based on brand, size, type, and specific function. Additionally, there is uncertainty about the fraction of the product that can be replaced by the resin and the extent to which the resin needs to be fractionated (at additional cost of manufacturing).

A combination of high price and moderate market size suggests the products with the highest economic potential (without accounting for processing costs for the raw material) are residential pesticides, paints and coatings, wood preservative coatings, amine-epoxy strippable coatings, and adhesives. These five most promising resin co-products were investigated further. Fig. 4 shows resin prices estimated according to the resin's function and mass fraction in each product. A distinction between conservative, baseline, and optimistic prices was made based on the range of prices found while surveying each product. The baseline estimates are based upon a single source value that represents a central estimate within the range. The conservative and optimistic prices are the minimum and maximum values of the prices found. Including conservative, baseline, and optimistic prices for each category conveys the uncertainty associated with these initial estimates.

Within Fig. 4, mass fractions of the resin were applied to the overall product prices to estimate the portion of the price that can be attributed to the resin. For pesticides, wood preservative coatings, and paints and coatings, resin accounts for 10–40 % of the total product composition (Table A5), assuming the insecticidal/anti-termitic function of the resin can entirely replace the typical fraction of active ingredients. For adhesives, resin accounts for 5–40 % of the total product assuming resin acts as the primary base resin in polyurethane, hot melt packaging, water born polyurethane, and pressure sensitive adhesive formulations (Strickland, 2013). For amine-epoxy strippable coatings, resin can account for up to 10 % of the total product as a plasticizer and maintain performance characteristics (Thames and Kaleem, 1991). Based on these application assumptions, guayule resin has an estimated price ranging from 0.1 to 6.8 USD/kg. Domestic pesticide use gave the highest specific price (1.1–6.8 USD/ kg), followed by wood preservative coatings (0.8–6.0 USD/kg), paints and coatings (0.7–6.0 USD/kg), and epoxy strippable coatings (0.8–2.5 USD/ kg). The specific price was lowest for adhesive applications (0.125–1.6 USD/kg). It is important to note that these price ranges do not account for the fractionization, processing, packaging, and distribution of the resin and/or final products. Further modeling and experimental work are required to fully understand the raw material value for resin.

6. Recommendations for guayule resin biorefining

The complexity of natural resin means that there can be no single technique for separation. Previous studies have concentrated on identifying the most efficient extraction method for specific groups of natural resin compounds based on chemistry. The next step for guayule resin commercialization is identifying a series of efficient separation techniques in a reliable chemical process model that can be used to inform economic and life cycle assessments. A potential guayule resin refinery model, with alternative pathways, is proposed in Fig. 5 based on previous characterization work (Cheng et al., 2020; Dehghanizadeh et al., 2020). This model was based on two scenarios: (1) freshly-harvested and (2) field-dried shrub. Output stream decision points are shown as diamonds, allowing multiple scenarios to be shown on a single diagram. Unit operation selection is based on three factors: a) the potential for scale up, b) the extraction efficiently for target compounds, and c) minimization of environmental impacts. Pretreatment steps, like parboiling, milling, grinding, de-leafing, stabilizing, hydrolyzing, and flaking, were not included in the model and would need to be considered in a final evaluation. Such pretreatment steps can and do strongly affect the efficiency of processing steps.

From Fig. 5, the first unit (110) for fresh shrub material is an essential oil (EO) extraction such as hydro-distillation or steam distillation. Non-volatile compounds from EO extraction can either be sent to a drying unit (130) or to a microwave-assisted extraction (MAE) separation unit (120), which can handle wet biomass after adjusting the moisture content of the solid-liquid mixture with a dewatering unit. Microwave-assisted extraction would remove the majority of guayule resin constituents (sesquiterpenes, triterpenes, phenolics, and fatty acids). Residues, rich in rubber and bagasse, would then be sent to the drying unit (130). This pathway is expected to slightly increase the rubber concentration in the non-volatiles residues and act as a pretreatment step, since the walls of the rubber-containing cells would be disrupted by microwaves. Due to the poor selectivity (presence of LMWR in acetone extracts and resin in hexane extracts) and environmental concerns associated with using organic solvents in conventional solvent extraction, SFE/PLE (units 140 and 150) would be used next as highly selective methods to separate the wide range of secondary metabolites, with rubber and bagasse being products after unit 140. A similar process using supercritical CO_2 extraction in separation, fractionation, and purification of rubber and resin was patented by Cornish et al. (2006).

The first scenario was developed to exploit the maximum amount and quality of terpenoids (e.g. α-pinene, β-pinene, caryophyllene, camphene, etc.). The yield and composition of major volatile compounds are strongly influenced by the drying process (Dong et al., 2011; Ghasemi Pirbalouti et al., 2013; Szumny et al., 2010). Similarly, some hydrocarbons are volatilized due to the heat produced during milling. Field-dried biomass has the advantage of being easily transported,

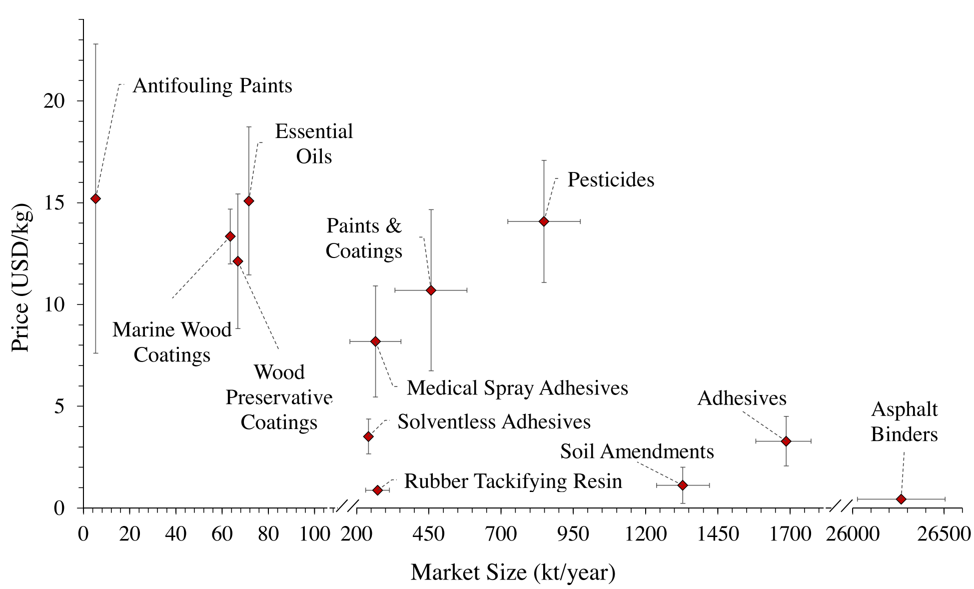


Fig. 3. Estimated prices of potential resin-containing products; uncertainty bars represent the range of prices reported in the references surveyed.

stored, and handled; on the other hand, freshly-harvested shrub has the advantages of avoiding the biochemical reactions and increased biological deterioration of aromatic plants that occurs during drying (Díaz-Maroto et al., 2003; Hamrouni-Sellami et al., 2013). The total amount of guayulins A–D in a fresh sample is approximately 15 % higher than in dried samples, a factor that cannot be ignored if applications of the resin sesquiterpenes are to be targeted (Spano et al., 2018). Assuming guayulins C and D are most likely derived from the oxidation/degradation of guayulins A and B, the concentration of guayulins C and D were significantly higher in the dried shrub (Martínez et al., 1986; Spano et al., 2018). Guayule leaves (green and dried) and flowers (peduncles and inflorescences), which contribute on average 32 % and 6 %, respectively, to the total biomass weight (Teetor et al., 2009) and are rich in resin (7–8 %), protein (12–22 %), and waxes (2.5 wt.% dry basis) (Banigan et al., 1982), are discarded prior to bulk rubber extraction. Wax extracted from guayule leaves has one the highest melting points (76 °C) recorded for a natural wax, giving it value-added potential as a source of hard wax (Banigan et al., 1951). Since guayule rubber is also synthesized and stored in the mesophyll cells of leaves (Madhavan and Benedict, 1984), the presence of leaves, which contain 0.5–2.0 % of total plant rubber (Gilliland and van Staden, 1986; Teetor et al., 2009), can increase the total rubber yield. Therefore, processing of fresh guayule shrub provides the opportunity to extract more terpenes, protein, and waxes than field-dried shrub.

Latex extraction (unit 300) from fresh shrub is another approach to rubber extraction since the bagasse produced from latex extraction is rich in resin (around 40 %) that may be a suitable feedstock for the SFE. Traditional resin extraction from resinous bagasse is difficult and inefficient because of the low percolation rate of solvents (Cornish et al., 2006). Supercritical CO_2 extraction (in unit 150) with high diffusivity properties has the potential to penetrate the low porosity bagasse cells and facilitate the release of compounds of interest at high percolation rates. Supercritical CO_2 with ethanol co-solvent can increase resin yield up to 12 % of total defoliated biomass compared with 7 % by acetone extraction (Punvichai et al., 2016).

The scenario using field-dried shrub, starting with unit 200, addresses the difficulties of using fresh shrub: fungal contamination, mold and bacteria growth, limitations caused by the presence of moisture on some extraction methods like SFE, and the presence of leaf material. (Leaf materials can reduce the rubber recovery due to the formation of very fine particles that clog the filtration systems and increase the ash content associated with rubber degradation and quality reduction (Battistel et al., 2017).) The bagasse produced from simultaneous extraction (unit 210) can be further processed with supercritical CO_2 to remove the residual solvent and extract value-added chemicals (pathway not shown). Key to the proposed dried shrub pathway is the multiple options for resin separation immediately after rubber processing, including SFE/PLE (unit 140), solvent extraction (unit 230) followed by SFE/PLE or terpene fractionation (unit 240), or saponification (unit 250) followed by solvent extraction or vacuum distillation (unit 260). Each pathway leads to similar groupings of resin products and could be tuned depending on the end product markets.

7. Conclusions

This review provides insight into the chemistry, extraction methods, and applications of guayule resin. The complexity and range of polarity of molecules in guayule resin mixtures (terpenoids, fatty acids, phenolics, steroids, alkaloid compounds, etc.) necessitate the evaluation of many methods for extracting constituents for value-added applications. Characterizing the distribution of chemical structures in guayule resin is a crucial step for finding the most appropriate extraction method. Modern extraction techniques reduce extraction time, energy, and solvent consumption, yet a completely “green” technique that enables isolation of all of the targeted compounds simultaneously is still elusive.

Guayule resin, often considered in the context of a byproduct of guayule rubber production, has the potential to overtake rubber value if the resin fractions can be suitably separated and used in high-value, medium-scale applications such as pesticides, paint, and coatings. Of the developed methods for natural resin fractionation with potential for scale-up, pressurized solvent extractions, like supercritical CO_2 and pressurized liquid extraction, are advantageous over other extraction methods due to the increased solubilizing power for the desired compounds and the higher solvent diffusivity. Care must be taken with higher extraction temperatures to avoid degradation of some high-value thermolabile compounds and biopolymers (rubber) into the resin extract phase. The potential of resin utilization for improving the economic viability of guayule as an industrial crop means that economic and lifecycle analyses of conceptual biorefineries need to include multiple pathways, starting from the whole plant.

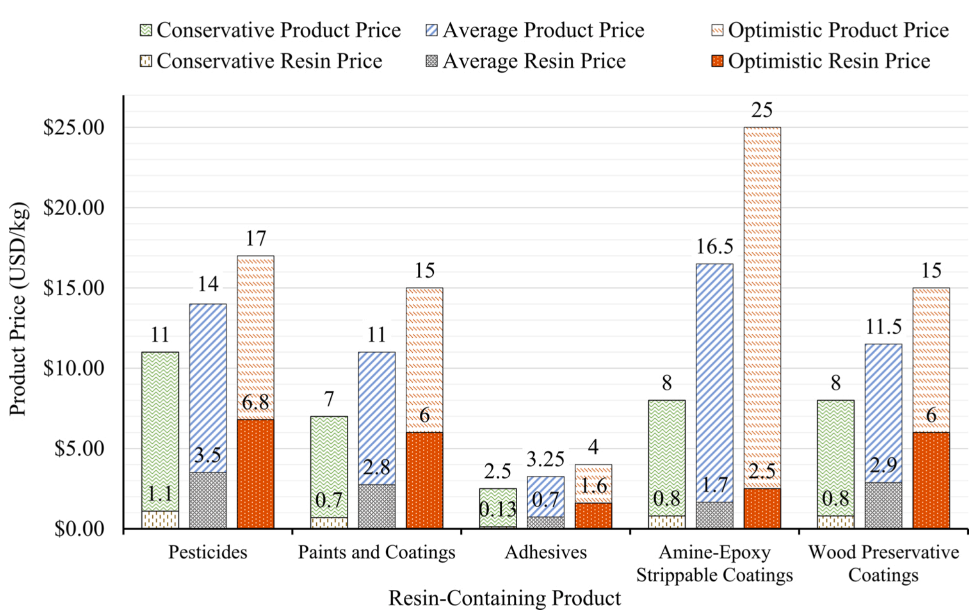


Fig. 4. Potential products and estimated resin prices based on the respective resin functions. Price bars are divided into product (lighter color) and the mass fraction that is assumed to be replaced with resin (darker color).

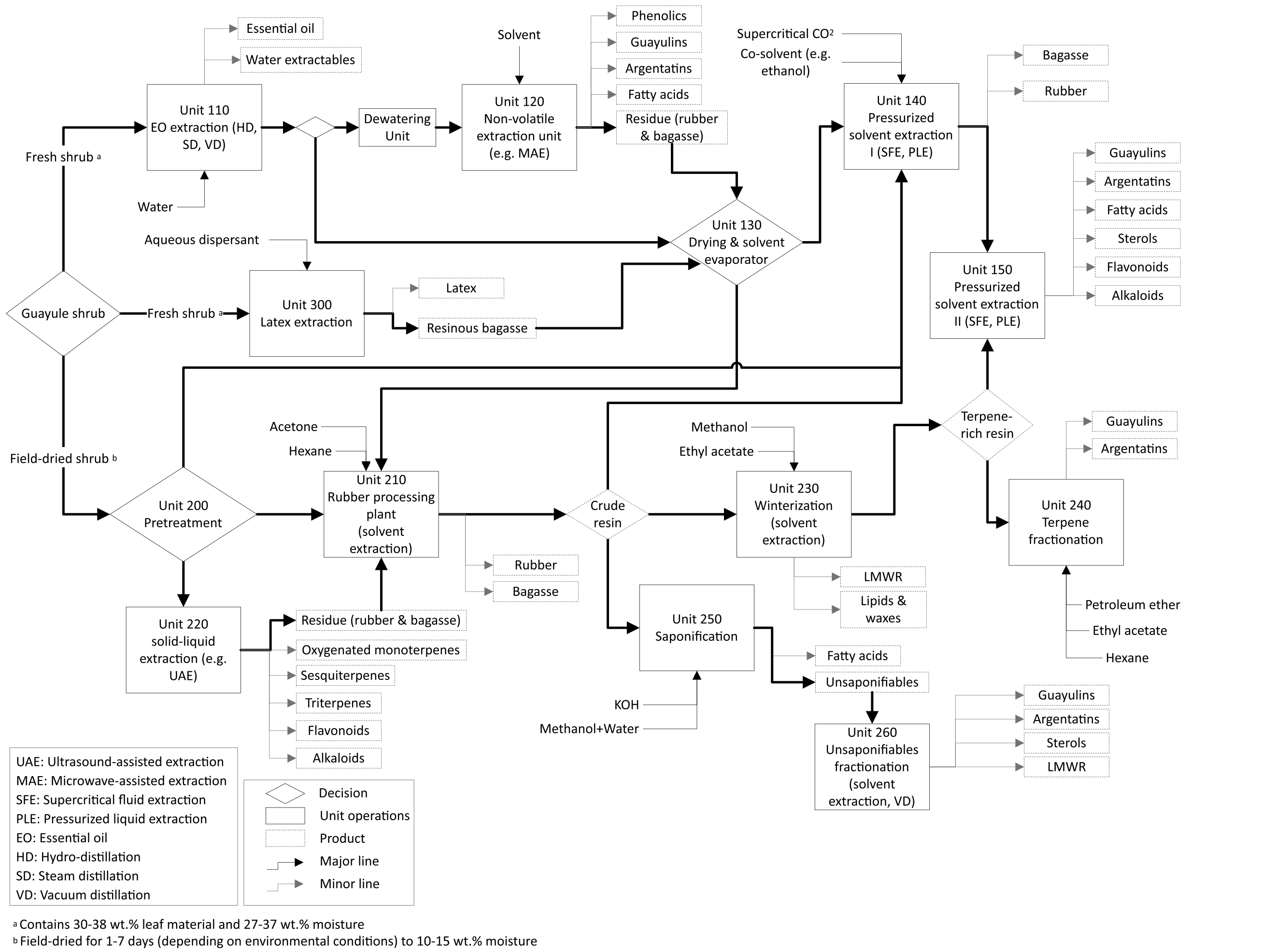


Fig. 5. A block flow diagram (BFD) of proposed guayule resin refinery processes.

Declaration of Competing Interest

The authors report no declarations of interest.

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Appendix A

Table A1
Guayule resin and rubber extractions using supercritical CO_2 extraction, summarized from Cornish et al. (2006) and Punvichai et al. (2016).

Sample	T (°C)	P (bar)	time (min)	Co-solvent (co-solvent/ feedstock ratio)	CO_2 flowrate	Extract yield (wt.%)	Results
1 [a]	60	345	30	–	3 (liters/ min)	2.9	High selectivity for resin
2 [a]	100	675	23	hexane (4/1)	3 (liters/ min)	8.0	Best extraction capability for rubber and lowest concentration of resin
3 [a]	100	345	45	hexane (22/3)	3 (liters/ min)	4.7	Good extraction capability for rubber and low Resin concentration
4 [a]	80	675	60	hexane (17/2)	3 (liters/ min)	11.2	Very good selectivity for rubber
5 [a]	100	675	60	1-hexane (17/2)	3 (liters/ min)	5.9	Very good selectivity for rubber
6 [a]	80	655	60	cyclohexane (17/2)	3 (liters/ min)	6.2	Very good selectivity for resin
7 [a]	100	655	60	iso-octane (17/2)	3 (liters/ min)	5.5	Good selectivity for resin
8 [a]	100	675	30	water (1/2)	3 (liters/ min)	4	High selectivity for resins and relatively low selectivity for rubber
9 [a]	60	345	24	water (1/2)	3 (liters/ min)	4.2	Concentration of resin is not nearly as high as the previous experiment. The yield is highest
10 [a]	9.2	138	–	–		–	No extract is collected

(continued on next page)

Table A1 (*continued*)

Sample	T (°C)	P (bar)	time (min)	Co-solvent (co-solvent/ feedstock ratio)	CO_2 flowrate	Extract yield (wt.%)	Results
					3 (liters/ min)		
11 [a]	60	345	45	acetone (1/1)	3 (liters/ min)	4.9	Extraordinary selectivity for resin.
12 [b]	40	250	60	acetone	34.4 (g/ min)	4.8	After 60 min increasing extraction time doesn't results in a visible gain. (Yield $_{120\ min}$ = 5.1 %)
13 [b]	33	250	60	acetone	34.4 (g/ min)	6.3	This yield is higher than the obtained yield (5.2 %) from ASE (ref. Method) using acetone
14 [b]	33	250	60	ethanol	34.4 (g/ min)	9.6	Ethanol clearly displays a better co-solvent power than acetone
15 [c]	33	250	60	ethanol	34.4 (g/ min)	10.1	Optimum flowrate of CO_2: 34-51 ml/min
16 [c]	33	200	60	acetone	34.4 (g/ min)	7.7	No significant changes in yield with increasing "P" from 200 to 300 bar using acetone as co-solvent
17 [c]	33	300	60	ethanol	34.4 (g/ min)	12.1	A constant increase in yield with increasing "P" from 100 to 300 bar using ethanol as co-solvent
18 [c]	60	250	60	ethanol	51.6 (g/ min)	12.3	Maximum yield is obtained between 45–60 °C using ethanol as co-solvent
19 [c]	60	250	60	acetone	51.6 (g/ min)	7.03	Co-solvent power of acetone is almost T independent over the tested range (33–80 °C)

[a] Feedstock: Entire fresh sample, chipped: 3/8", dried at 80 °C.
[b] Feedstock: defoliated shrub, vacuum-dried at 40 °C for 3 days. Moisture <10 %. Harvest place: France.
[c] Feedstock: Bagasse obtained after water-based extraction. Oven dried at 40 °C. Ground 0.5 mm. Harvest place: Spain.

Table A2
Guayule resin and rubber extraction using ASE (accelerated solvent extraction).

Sample	Solvent	Cycle/time (min)	T (°C)	Resin yield (wt.%)	Solvent	Cycle-time (min)	T (°C)	Rubber yield (wt.%)	Remark	Reference
1 [a]	acetone	repeatedly/30	100	7.2	–	–	–	–	–	Pearson et al. (2013)
2 [a]	acetone	three/20	100	9.4	cyhex	three/20	140	4.0	–	
3 [a]	acetone	two/5	40	7.4	cyhex	three/20	140	5.3	–	
4 [b]	acetone	three/5	100	8.0	hex	three/20	140	2.3	0 h. pre-extraction heating time at 200 °C	Cornish et al. (2013)
5 [b]	acetone	three/5	100	3.2	hex	three/20	140	0.1	12 h. pre-extraction heating time at 200 °C	
6 [c]	acetone	three/5	100	12.4	hex	three/20	140	0.8	1 h. pre-extraction heating time at 150 °C	
7 [c]	acetone	three/5	100	12.6	hex	three/20	140	0.8	0.3 h. pre-extraction heating time at 200 °C	
8 [d]	acetone	three/5	100	9.4	cyhex	three/20	140	7.1	48 h. pre-extraction heating time at 50 °C	
9 [d]	acetone	three/5	100	9.5	cyhex	three/20	140	7.6	24 h. pre-extraction heating time at 75 °C	
10 [e]	acetone	three/5	100	5.5	cyhex	three/20	140	6.9	4 h. pre-extraction heating time at 75 °C	
11 [f]	acetone	three/5	100	16.2	cyhex	two/20	100	1.5	–	Salvucci et al. (2009)
12 [g]	acetone	two/20	24	8.8	cyhex	two/20	100	7.3	air-dried 4 days at 23 °C	
13 [g]	acetone	two/20	24	6.2	cyhex	two/20	100	8.4	air-dried 4 days at 23 °C	
14 [h]	acetone	three/20	40	9.3	hex	three/25	120	9.05	–	Suchat et al. (2013)
15 [i]	acetone	three/20	40	10.7	hex	three/25	120	5.8	–	Taurines et al. (2019)
16 [j]	acetone	three/20	40	9.3	hex	three/25	120	9.2	–	

[a] 2-year old shrub, AZ2 line, hand-harvested, foliated, dried at 65 °C.
[b] bagasse from latex extraction of 3-4-year-old shrub in aqueous hydroxide, partially defoliated, dried outdoors at AMT, ground for 90 s using 2-mm mesh screen.
[c] 2-year old shrub, AZ2 line, hand-harvested, foliated, dried at 50 °C, ground for 90 s using 2-mm mesh screen.
[d] Three-year old shrub, defoliated, harvested in summer.
[e] Three-year old, collected during autumn.
[f] Guayule stem tissue with high latex rubber content (~9.6 %), harvested from plant grown under 9 h, 16°C light/15 h, 7°C dark photoperiod, freeze-dried at -80 °C.
[g] Two-year old shrub, AZ2 line, defoliated, ground to 1.7 mm, latex content.5.3 %.
[h] 1-2-year-old shrub, harvested in Spain from AZ2, AZ1, 11595, N565, 593, and CAL-6 lines, leaves and flowers, partially dried at AMT for two days, ground to <0.5 mm, vacuum dried at 40 °C for 48 h.
[i] ~ 4-year-old shrub, line CL1 (parent 11591, triploid), leaves and flowers removed, dried at 70 °C for 15 h, freeze dried at -80 °C for 15 h, ground to 1 mm.
[j] ~ 4-year-old shrub, line CLA1 (parent AZ 101, tetraploid), leaves and flowers removed, dried at 70°C for 15 h, freeze dried at -80 °C for 15 h, ground to 1 mm.

Table A3
List of potential products and the corresponding economic value.

Product	Value Range (USD/kg)	Source	Item
Asphalt binder	0.26-0.61	The Ohio Department of Transportation, 2019	
		Poten and Partners Asphalt Weekly Monitor 2017	
		Colorado Asphalt Pavement Association 2012-Present	
		Idaho Department of Transportation Asphalt Price Index 2020	
		Oregon Department of Transportation Monthly Asphalt Cement Material Price 2019	
		Louisiana Department of Transportation and Development 2019	
		New Mexico Department of Transportation 2019	
		Arizona Asphalt Price Index 2019	
Adhesives	1.30- 9.03	American Floor Mats	Rubber Flooring Adhesive Item No: STADHRFA
		Zoro	Roberts #3000-4 ZORO #: G9189680
		ACE Hardware	Zinsser SureGrip 122 High Strength Adhesive 1 gal.
		Zoro	Rust-Oleum #02880 Zoro #: ZORO #: G9489234
		Zoro	Catchmaster #BG-1 ZORO #: G3279245
		Zoro	Weldwood #00142 ZORO #: G2310366
		Lumber Liquidators Flooring Company	Pro Bond Adhesive 5 Gallon SKU: 10047804
		Lumber Liquidators Flooring Company	Ultrabond eco 995 Adhesive 5 Gallons SKU: 10039742
		Zoro	Roberts #1407-1 ZORO #: G5123727
		Zoro	Roberts #2057-4 ZORO #: G9189568
		Zoro	Roberts #2057-1 ZORO #: G5118233
Solventless (laminating) adhesives	2.07- 7.88	The Home Depot	Roberts 4 Gal. Multi-Purpose Carpet and Sheet Vinyl Adhesive (24 Pail Pallet) Internet #202935530
		EMI Supply	Titebond Solvent Free Multi-Purpose Flooring Adhesive, 1 Gallon Can
		The Home Depot	Roberts 4 Gal. Premium Vinyl Tile Glue Adhesive (24 Pail Pallet)
		The Home Depot	Henry 4.75 Gal. 203 Cold Applied Roof Adhesive
		The Home Depot	Roberts 4 Gal. Resilient Flooring Adhesive for Fiberglass Sheet Goods and Luxury Vinyl Tile
		The Home Depot	Custom Building Products SimpleSet Gray 1 Gal. Premixed Thin-Set Mortar
		The Home Depot	Titebond III Ultimate Wood Glue (2-Pack)
		The Home Depot	OmniGrip 3-1/2 Gal. Maximum Strength Adhesive
		The Home Depot	Custom Building Products AcrylPro 3-1/2 Gal. Ceramic Tile Adhesive (24 buckets/pallet)
		The Home Depot	Roberts 1 Gal. Universal Flooring Adhesive
Medical spray adhesives	5.45- 10.87	Reportlinker. "The Laminating Adhesives Market Is Projected to Register a CAGR of 6.9 %, in Terms of Value, between 2019 and 2024." *PR Newswire: Press Release Distribution, Targeting, Monitoring and Marketing*, 21 Oct. 2019, www.prnewswire.com/news-releases/the-laminating-adhesives-market-is-projected-to-register-a-cagr-of-6-9-in-terms-of-value-between-2019-and-2024–300941835.html.	
		"Spray Adhesives Market." *Market Research Firm*, Markets and Markets, Jan. 2018, www.marketsandmarkets.com/Market-Reports/spray-adhesive-market-201133268.html.	
Rubber tackifying resin	0.75- 2.09	Southern Geo Supply	Profile Tornado Tack ST-1000 Straw Tackifier, 50 lb Bag SKU: ST-1000
		Southern Geo Supply	Profile Tacking Agent 3, Tackifier and Dust Control SKU: LF21TACK3
		Alibaba	Rubber Chemicals Petroleum Resin Tackifier C5 CAS- No: 64742-16-1
		Alibaba	Rubber Chemicals Petroleum Resin Tackifier C5 CAS- No: 64742-16-2
Strippable coatings	9.68- 27.18	C&C Industrial Sales	BINKS Part No: 29-249 Booth Coat (White)
		TOOLiD	3M® 6840 - 5 Gal. Booth Coating Item # mpn1453059886
		Zoro	Precision Brand #43125 ZORO #: G7065307
		MSC Direct	Clear Water Base Booth Coating Part #:00240069
		Collisions Services	Klean-Strip Mask & Peel Spray Booth Coating CMP229 SKU #1700850
Wood preservative coating & Marine wood coating	2.90- 17.16	The Home Depot	Olympic Waterguard 1 gal. Clear Multi-Surface Waterproofing Sealant SKU #1003317178
		Amazon	Rust-Oleum 1904A Wolman (Woodlife) CopperCoat Green Wood Preservative-Below Ground, Quart 1904A
		Amazon	Rust-Oleum Clear 902 Wolman Classic Wood Preservative-Above Ground, Quart Part Number 902
		Amazon	Watco 68141 Wipe-On Polyurethane Finish, Quart, Clear Satin Part Number 68141
		ACE Hardware	Flood CWF-UV Matte Redwood Water-Based Wood Finish 5 gal. Item no.1465160
		Amazon	DEFY Extreme 5 Gallon Semi-Transparent Exterior Wood Stain, Natural Pine Part Number 300163
		East Coast Hardware	Twp 1500 Series Twp-1501-1 Wood Preservative, Cedartone, 1 Gal Can SKU# 5111515
		Amazon	Gemini 204082 TWP1500-1 1 G Clear Wood Preservative Part Number 204082
		Green Building Supply	Vermont Natural Coatings, Exterior, Penetrating Water Proofer Item #42075
		Western Log Home supply	X-100 Natural Seal Wood Protective for Log Homes Item #: NSWP5
		Walmart	SEAL-ONCE MARINE Penetrating Wood Sealer, Waterproofer & Stain (1 Gallon). Part Number SO7614
		ACE Hardware	Rust-Oleum Marine Coatings Outdoor Gloss White Marine Topside Paint 1 qt. Item no.16754
		Cabela's World's Foremost Outfitter	Parker Coatings Gallon Duck Boat Paint Item: IK-423722
		Lowe's	Rust-Oleum RockSolid Tintable Resurfacer (Actual Net Contents: 116-fl oz) Item # 1030528
		Amazon	

(*continued on next page*)

Table A3 (*continued*)

Product	Value Range (USD/kg)	Source	Item
			Penofin F3emaga Marine Oil Transparent Oil-based Wood Finish, 1 Gallon Part Number F3EMAGA
		Jamestown Distributors	TotalBoat TableTop Epoxy
		Amazon	Waterlox Original Marine Sealer- Gallon by Waterlox Part Number TB 3809 Gallon
		Wholesale Marine	Pettit Flagship Varnish SKU:PET-2015 G.1
		Wholesale Marine	Interlux Schooner Gold Marine Varnish QT SKU:ILX-YVA-500Q
		Westmarine	PETTIT PAINT–SeaGold Marine Wood Treatment, Quart Model # 17977778
		Jamestown Distributors	TotalBoat Lust Marine Varnish
		Wholesale Marine	Interlux Sikkens Cetol Marine Wood Finish SKU:ILX-IVA-300.1
Pesticides	11.08–17.10	DoMyOwn	Garden Tech Sevin Insect Killer Concentrate UPC 613499010148
		Mosquito Controls	Malathion 5EC Insecticide, Drexel
		Mosquito Controls	Malathion 57EC, FMC Corporation
		Mosquito Controls	Malathion 5 EC Insecticide, Winfield
		Solutions Pest & Lawn	Imidacloprid 2 F Insecticide QUALI-PRO
		Do It Yourself	Permethrin SFR Insecticide 36.8 % Control Solutions
		DoMyOwn	Monterey B.t. Insecticide UPC 022179103900
Soil amendments	0.24- 1.72	Amazon	Aries Green Biochar Soil Amendment 5-Gallon Plastic Bucket – USDA, IBI Certified – 100 % Biochar ADIN B07V9VT3MK
		eBay	Rogue Valley Premium Biochar (10 Cu. Yds) Item Number 322550315092
		Oregon Biochar Solutions Pricing	
		Oregon Biochar Solutions Pricing	
		eBay	Biochar produced from sustainable biomass (ARTichar) - 55 gallons item number:272304233958
		East Coast Hardware	Premier 0128 P Peat Moss, 2.2 Cu-Ft Bale SKU# 5100155
		BioBizz	BioBizz - Light-Mix 50 Liter Bag (60/Plt) SKU: 722997
Essential oils	11.45-18.72	"U.S. Essential Oil Market Size, Share: Industry Analysis Report, 2024." *U.S. Essential Oil Market Size, Share | Industry Analysis Report, 2024*, Grand View Research, Feb. 2019, www.grandviewresearch.com/industry-analysis/us-essential-oil-market.	
		Duncan, Eric. "Topic: Essential Oils." *Www.statista.com*, M. Shahbandeh, Aug. 2019, www.statista.com/topics/5174/essential-oils/.	
		"Essential Oils Market Size, Growth, Share: Global Report 2026." *Essential Oils Market Size, Growth, Share | Global Report 2026*, Market Research Report, July 2019, www.fortunebusinessinsights.com/industry-reports/essential-oils-market-101063.	
		IBISWorld, Inc. "Industry Market Research, Reports, and Statistics." *IBISWorld*, clients1.ibisworld.com/reports/us/industry/productsandmarkets.aspx?entid = 5061.	

Table A4
Product Market Size Data.

Product	Market Size	Reference
Asphalt Binder	IBIS World Market Report	https://clients1.ibisworld.com/reports/us/industry/majorcompanies.aspx?entid=450
	National Asphalt Pavement Association	http://www.asphaltpavement.org/index.php?option=com_content&view=article&id=891
	AsphaltRoad.org	http://www.asphaltroads.org/assets/_control/content/files/Asphalt_White_Paper_doc.pdf
	National Asphalt Pavement Association	http://www.asphaltpavement.org/index.php?option=com_content&view=article&id=14&Itemid=33
Adhesives	IBIS World Market Report	https://clients1.ibisworld.com/reports/us/industry/productsandmarkets.aspx?entid=493
	Freedonia Group	https://www.freedoniagroup.com/industry-study/adhesives-sealants-3257.htm
	Market Watch	https://www.marketwatch.com/press-release/adhesives-and-sealants-market-opportunity-2018-2026-detailed-in-new-research-report-2019-01-24
	GlobeNewswire Reports and Data	https://www.globenewswire.com/news-release/2019/08/13/1901231/0/en/Adhesives-and-Sealants-Market-To-Reach-USD-88-25-Billion-By-2026-Reports-And-Data.html
	Grand View Research	https://www.grandviewresearch.com/industry-analysis/adhesives-and-sealants-market
Solventless (laminating) adhesives	PR Newswire	https://www.prnewswire.com/news-releases/the-laminating-adhesives-market-is-projected-to-register-a-cagr-of-6-9-in-terms-of-value-between-2019-and-2024–300941835.html
Medical Spray Adhesives	PR Newswire	https://www.prnewswire.com/news-releases/the-laminating-adhesives-market-is-projected-to-register-a-cagr-of-6-9-in-terms-of-value-between-2019-and-2024–300941835.html
	Markets and Markets	https://www.marketsandmarkets.com/Market-Reports/spray-adhesive-market-201133268.html
Rubber Tackifying resin	Market Watch	https://www.marketwatch.com/press-release/global-tackifiers-market-size-is-projected-to-be-around-us-4760-million-by-2024-2019-04-12
	Maximize Market Research	https://www.maximizemarketresearch.com/market-report/global-tackifier-market/27433/
	Ink Wood Research	https://www.inkwoodresearch.com/reports/north-america-tackifiers-market-forecast-2016-2024/
Paints and coatings	Statista	https://www.statista.com/statistics/684695/united-states-paint-and-coatings-demand-by-market/
	Grand View Research	https://www.grandviewresearch.com/industry-analysis/paints-coatings-market
Marine coatings	Industry Arc	https://www.industryarc.com/Report/11726/marine-coatings-market.html
	Coatings World	https://www.coatingsworld.com/issues/2018-08-01/view_features/marine-coatings-market-145178/
	Coatings World	https://www.coatingsworld.com/issues/2018-08-01/view_features/marine-coatings-market-145178/
	PR Newswire	

(continued on next page)

Table A4 (*continued*)

Product	Market Size	Reference
		https://www.prnewswire.com/news-releases/marine-coatings-market-to-burgeon-at-6-1-cagr-thanks-to-rise-in-ship-building-activities—tmr-300860524.html
	Statista	https://www.statista.com/statistics/1062762/global-marine-coating-market-value/
Wood preservative coatings	Market Watch	https://www.marketwatch.com/press-release/wood-coatings-market-2019-global-industry-share-demand-top-players-industry-size-future-growth-by-2023-market-reports-world-2019-05-09
	Coatings World	https://www.coatingsworld.com/issues/2016-02-01/view_features/wood-coatings-market-757548/
	Tec Navio	https://www.technavio.com/report/global-wood-coatings-market-analysis-share-2018
Antifouling Paints	Kake News	http://www.kake.com/story/41139819/antifouling-coating-market-share-size-2019-industry-growth-factors-top-leaders-development-strategy-future-trends-historical-analysis-competitive
	PR Newswire	https://www.prnewswire.com/news-releases/marine-anti-fouling-coatings-worldwide-market-insights-2019——hull-coatings-segment-to-dominate-the-market-300883344.html
	Grand View Research	https://www.grandviewresearch.com/industry-analysis/antifouling-coating-market
	IBIS World Market Report	https://clients1.ibisworld.com/reports/us/industry/productsandmarkets.aspx?entid=484
Pesticides	Ag Professional	https://www.agprofessional.com/article/study-shows-global-pesticide-market-reach-81-billion-five-years
	Pesticide Action Network North America	http://www.panna.org/blog/us-and-world-pesticide-use
	Grand View Research	Https://www.grandviewresearch.com/industry-analysis/essential-oils-market
Essential Oils	Statista	https://www.statista.com/topics/5174/essential-oils/
	Fortune Business Insight	https://www.fortunebusinessinsights.com/industry-reports/essential-oils-market-101063
	IBIS World Market Report	https://clients1.ibisworld.com/reports/us/industry/productsandmarkets.aspx?entid=5061

Table A5
Typical fractions of active ingredients contained in pesticide products.

Product Category	Active Ingredient Percentage	Source	Item
Termiticide, Insecticide and Fungicide	40 %	Forestry Distributing, North America's Forestry Leader	Niscus Corportation Bora-Care Termiticide, Insecticide & Fungicide, Nisus
Wood Preservative coatings	25 %	Perma-chink Systems, Inc.	Shell Guard Concentrate Borate Glycol Wood Preservative Coating
Termiticide and Insecticide	36 %	DoMyOwn	Tengard SFR Termiticide Insecticide
Insecticide	21 %	Solutions Pest & Lawn	CSI Dominion 2 l Insecticide
Termiticide and Insecticide	9 %	Do-It-Yourself Pest Control	Control Solutions Taurus SC Insecticide-Termiticide

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