

Review

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Review

Strategies for the Transformation of Waste Cooking Oils into High Value Products: A Critical Review

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Abstract: Waste Cooking Oils (WCOs) ARE produced in large quantities worldwide from hospitality, household and other industrial compartments. Today many Countries have no specific legislation regarding WCOs management, generating a crucial environmental problem. Presently WCOs are mainly employed by industry as feedstock for biodiesel and energy production. Nevertheless, the use of WCOs as a primary feedstock for second generation biodiesel production depends on its availability, and often import of biodiesel or WCOs from other countries is required. Additionally, the EU is pushing towards the privileged use of biowaste for alternative high value products, other than biodiesel, to reach carbon neutrality by 2050. Thus, the aim of this review is to give an overall comprehensive panorama of the production, impacts, regulations and restrictions affecting WCOs, and their possible uses to produce high value materials such as bio lubricants, bio surfactants, polymers and polymer additives, road and construction additives, bio solvents among others. Interestingly many reviews have been reported in the literature addressing the use of WCOs for the preparation of a specific class of polymer, but a general comprehensive review on the argument is missing.

Keywords: waste cooking oil; recycling; polymer synthesis; circular economy

1. Introduction

Demand and consumption of vegetable oils have significantly increased in the last 15 years, moving from 83 Miot in 2000, to over 217 Miot in 2023, of which 167 Miot were employed for biodiesel production, while the remaining part was used primarily by the food, feed, and pharma industry [1,2].

Globally, palm and soybean oils dominate the market with respectively 40.0% and 29.0% of the share, while other vegetable oils are produced in smaller quantities and include sunflower (9.7%), rape (6.3%), peanut (4.1%), corn (2.1%), coconut (1.9%), olives (1.8%) and sesame oil (1.1%) [2] (Figure 1a). Asia satisfies over 54.0% of worldwide vegetable oils demand, and in particular Indonesia is the world's greatest producer, followed by China, Malaysia, USA and Brazil (Figure 1b), [3–9]. Although biodiesel may be considered an attractive alternative for the partial substitution of fossil-based fuels, edible oils are an essential source of nutrition accounting for approximately 10.0% of the global average caloric food supply, second in importance only to cereals [10].

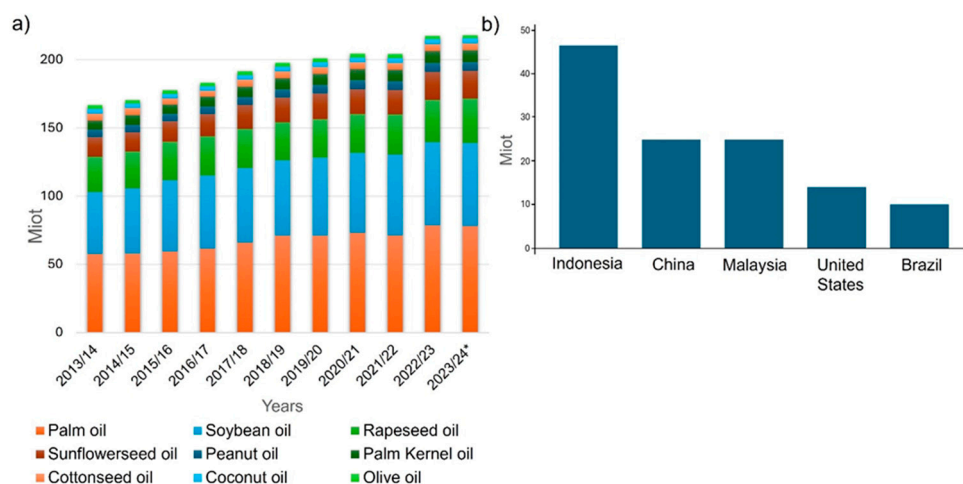


Figure 1. a) source of Vegetable oils and b) main geographical areas of production (Miot/year).

Additionally, it has been demonstrated that a correlation exists between the rise in biodiesel consumption in the USA and that of palm oil plantations in Southeast Asia, consequently increasing deforestation and global warming [11]. Thus, high production costs, threat for the food and feed value chain, together with socio-environmental impacts have made the use of edible oils as feedstock for biodiesel less appealing over time [12–14].

Alternative valuable feedstocks for biodiesel production are known as waste cooking oils (WCOs), fats, and grease together with more recent technologies starting from algae [1,15,16].

The term WCOs refers to exhausted cooking oils coming from hospitality and industrial sectors, no longer suitable for human consumption [13]. If disposed of in drains or sewers, WCOs form products that linger in the environment for many years, generating odors and problems in wastewater treatment plants, polluting soil and water courses, causing highly negative environmental impacts [17–19]. Additionally, WCOs may contain intermediate products formed by oxidation or microbial degradation which are toxic for microorganisms, algae, plankton, mussels, and amphipods [20,21], but also for humans causing serious illnesses such as dyspepsia, stomach-ache, diarrhea or gastric cancer [22]. The reuse and recycling of WCOs therefore opens new frontiers, reducing disposal costs and environmental impacts derived [23].

Presently WCOs are mainly employed at industrial level to produce biofuels and energy [24–35], yet in consideration of the EU requirements to reach carbon neutrality by 2050, the use of WCOs and in general of by-products and waste as fuels should be disfavored compared to their valorization for the production of high value chemicals [15,36,37]. In fact, the need to recover and recycle by-products or end of life products, reducing primary resource consumption and CO₂ emissions is becoming increasingly urgent, leading to a change from a linear economy based on a “take-make-discard” model, to a circular economy based on green chemistry and eco-design [38–41].

Alternative applications of WCOs include the production of bio lubricants [31,42–44], bio solvents [45], animal feed [46–49], asphalt additives [50,51], composite materials [52,53], polymers [54,55], fine chemicals production [56–58], non-aqueous gas sorbent devices [59,60], among others.

Within this panorama, the present review intends to give a general overview on the reuse of WCOs to produce high value products (polymeric materials), other than fuel and energy, published in the last ten years. Data available in the literature will be subdivided according to different application areas. Interestingly many reviews have been reported in the literature addressing specific topics on the use of WCOs for the preparation of a specific class of polymer, but a general comprehensive review of the argument is missing.

2. Methods

Systematic research of the existing literature was carried out thoroughly analyzing the current status of WCOs production, recycling techniques and valorization, EU directives and legislation on WCOs recovery and reuse, together with impacts derived. A combination of different keywords such as “waste cooking oil”, “used cooking oil”, “bio lubricant”, “bio surfactant”, “bio solvent”, “polyurethane”, “bio polymer”, “non isocyanate polymer”, “acrylic polymer”, “alkyd ester”, “epoxy resin”, “asphalt/construction additive, rejuvenating, anti-aging”, “circular economy”, “EU directives”. Specifically, one or a combination of two keywords were chosen for the selection of the papers and only those published in English language on peer reviewed journals were reported.

Research papers included in the review were collected through Web of Science Scopus, Google Scholar, ScienceDirect, ResearchGate, and were published between 2014 and 2024. In order to find the most updated data and regulations, we also searched websites of relevant organizations, such as European commission portal [61], and European Law portal [62], the European Commission press corner [63], the European Environment Agency (EEA) [64], the European Chemicals Agency (ECHA) [65], Statista [66] and Eurostat [67]. Once relevant sources were identified, references were reviewed for additional relevant information. This review focuses on WCOs reuse for the production of polymeric materials.

3. WCOs Production and Market

Although a large number of information are known on the production of vegetable oil worldwide (see Figure 1a, 1b), it is rather difficult to find accurate data quantifying the amounts of WCOs produced and available for valorization. Teixeira and co-workers reported that about 320 Kg of WCOs are produced for every ton of oil employed for cooking, with a valorization rate between 75% to 23%, according to the geographical area [18]. A rather worrying fact that emerges from the analysis by Teixeira is that out of 23 states evaluated, over 85% had no specific legislation regarding WCOs management. This data was also confirmed by a more recent paper by Zhao and coworkers indicating a general static trend over the last years in the adoption of more sustainable management strategies of WCOs, where the main alternative remains inadequate disposal in the environment [68].

In the USA, according to the Environmental Protection Agency, nearly 11.4 Miot of WCOs are collected from restaurants yearly, only small percentages of which are adequately managed [69], while production estimates are of about 15.0 Miot of WCOs [70]. Even the virtuous EU has a long way to go, considering that according to estimations, WCOs pro-capita production in the EU is of 8L/year, which considering a population of around 500 million people corresponds to a WCOs yearly production of 4.0 Miot. This value, which is four times higher than the currently collected amount, clearly evidences the gap between WCOs produced and collected [43]. Another possible estimation of WCOs yearly production in the EU can be done starting from EU yearly consumption of vegetable oils (24.0 Miot) and considering that according to Teixeira 32% is discarded, WCOs yearly production in EU could be even higher (around 7.7 Miot) [18]. This also accounts for the great difference between vegetable oils consumption (220.0 Miot), and WCOs collected worldwide (about 50.0 Miot) [69], distributed geographically as reported in Figure 2.

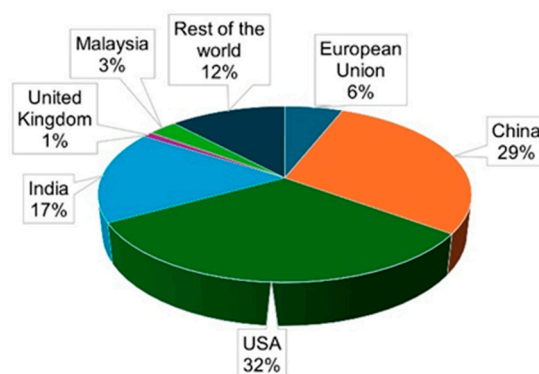


Figure 2. Geographical distribution of WCOs production worldwide.

Presently WCOs are mainly employed by the industry as feedstock for biodiesel and energy production [71–79]. In fact, it is well known that WCOs can be used to produce Fatty Acid Methyl Esters (FAME) through alkali catalyzed transesterification, and used as biodiesel [34,54,80,81]. Other processes for the valorization of WCOs are known such as cracking or hydrocracking, pyrolysis, and gasification processes for the production of biofuels [75]. Additionally, energy production by WCOs combustion has been reported in the literature, in different conditions, and with WCOs having different viscosity and acidity [82,83].

It should nevertheless be considered that although the use of WCOs can reverse the noncompetitive price of biodiesel from virgin biomass and reduce environmental negative impacts [84,85], however, the use of WCOs as a primary feedstock for second generation biodiesel production depends on its large-scale availability, and since local food waste is often insufficient, import of biodiesel or WCOs from other countries is required [86–88]. As evidenced by various LCA studies, import of WCOs is not a sustainable practice to guarantee energy supply, neither it contributes to reduce gas emissions [71,89]. In this concern, the EU renewable energy directive recently approved (Directive (EU) 2023/2413) states that “the share of biofuels and bioliquids, as well as of biomass fuels consumed in transport, where produced from food and feed crops, shall be no more than one percentage point higher than the share of such fuels in the final consumption of energy in the transport sector in 2020 in that Member State, with a maximum of 7 % of final consumption of energy in the transport sector in that Member State” [90]. Within this panorama alternative use of WCOs for high value products should be privileged in alternative to their use as biodiesel. Thus, in this review, no further discussion on the use of WCOs for biodiesel production is reported.

4. EU Regulations and Restrictions Affecting WCOs

As far as the EU is concerned, starting from the 1970s, with the waste oil directive 75/439/EEC, the European Commission committed to collecting used oils, limiting environmental hazards, and promoting recovery and recycling technologies [43,91].

During the last decade many regulatory updates have been implemented at EU level for the classification, storage, recovery or disposal of waste oils from hospitality, household and other industrial compartments. Amending Directive 2008/98/EC and European Commission (EC) Decision 2000/532/EC, EC Decision 2014/955/EU of 18 December 2014 [92] reports a harmonized list of wastes, continuously updated based on new knowledge and research studies. It should nevertheless be stressed that the inclusion of a material in this list does not necessarily imply that the material is a waste in all circumstances. Materials are considered to be waste only when the definition of waste in Article 1(a) of Directive 75/442/EEC is met [93].

Different wastes are defined by a six-digit code (Directive 2014/955/EU) [92] where the first two digits identify the manufacturing compartment generating the waste, the second two, processing operations, and the last ones a specific material. This implies that a waste may be identified by different EWC according to the manufacturing process which generates it, and vice versa specific

production units may need to classify their wastes in several chapters. As regards WCOs, they are classified as 200125 where 20 corresponds to “municipal wastes (household waste and similar commercial, industrial and institutional wastes)”, 01 to “separately collected fractions”, 25 to “edible oil and fat”. Edible oils are also found as mixtures with grease coming from wastewater treatment plants and are identified by EWC 190809, while if coming from agriculture, horticulture, aquaculture, forestry, hunting and fishing, food preparation and processing wastes the EWC is 0203.

Additionally, WCOs classified under EWC 200125 code are not hazardous for human health, but their inadequate disposal makes them potentially harmful for the environment. For this reason, Art. 1 of Directive 2008/98/EC laid down the “measures to protect the environment and human health by preventing or reducing the adverse impacts of waste mismanagement and improving the efficiency for reuse”, requiring Member States to adopt waste management plans [94]. As in other occasions the need to promote the development of a unified EU policy program, preserving Member State sovereignty, to adopt different approaches to waste management, recycling and disposal, inevitably creates logistic barriers across borders [36].

Considering that in 2018 still many Member States had not yet developed the necessary waste management infrastructures, EU Directive 2018/851 was issued amending EU Directive 2008/98/EC to “prevent the creation of structural overcapacities for the treatment of residual waste” and “improve the efficiency of resource use and ensuring that waste is valued as a resource, can contribute to reducing the Union’s dependence on the import of raw materials and facilitate the transition to more sustainable material management and to a circular economy model”(Directive (EU) 2018/851) [95]. This Directive among many others, sets out the EU legislative framework pushing towards the achievement of the EU Action Plan, the Sustainable development goals of the UN Agenda 2030 and achieving carbon neutrality by 2050. It is of fundamental importance for the development of new sustainable processes based on the circular economy model that a congruent and unified legislative framework should be adopted within the EU to allow development of new processes to transform waste in secondary primary materials, promoting neat-zero waste processes [31,43,45,96].

In this connection, the End of Waste directive EU 2008/98 is a fundamental milestone, determining necessary criteria to transform a waste into a valuable secondary primary material [94,97]. In particular, Directive 2008/98/EC defines “recovery” as “any operation the principal result of which is waste serving a useful purpose by replacing other materials which would otherwise have been used to fulfil a particular function, or waste being prepared to fulfil that function, in the plant or in the wider economy” [97]. Additionally, “recycling” is defined as “any recovery operation by which waste materials are reprocessed into products, materials or substances whether for the original or other purposes not including energy ... fuels or for backfilling operations”.

Specifically, requirements for “End of waste status” are that a waste shall cease to be waste if: (i) it has been demonstrated that “the substance is commonly used for specific purposes”; (ii) “a market or demand exists for such a substance or object”; (iii) “the substance fulfils the technical requirements for the specific purposes and meets the existing legislation and standards applicable to products” and (iv) “the use of the substance will not lead to overall adverse environmental or human health impacts”.

5. Environmental Impacts of WCOs

The environmental impacts derived from the inadequate disposal of WCOs are numerous and derive from accidental or deliberate release, causing environmental and biological alterations. WCOs highest environmental impact occurs when discharged into water bodies, or indirectly, if introduced into the sewer system [98–100]. WCOs are immiscible with water and generate a thin film, reducing oxygenation, limiting light filtration, affecting the life of aquatic plants and living beings. It is estimated that 1 kg of used vegetable oil can be evenly distributed to cover an area of 1000 m² [18]. Similarly, WCOs tend to form a hydrophobic film around the particles of earth and on the surface of plant roots creating a barrier to water and, consequently, precluding the intake of nutrients. Moreover, if WCOs penetrate the ground, they may reach wells of drinking water, making it

unsuitable for human consumption [99]. For this reason, it is essential to take appropriate precautions to protect groundwater and wells, but also surface waters that may be in contact with the deeper water reservoirs. Moreover, like synthetic oils and fossil-based products, WCOs can cover the surfaces of living organisms (for example birds and aquatic animals) forming an insulating film, decreasing their ability to breath, exchange heat and in some cases move.

The great risks associated with improper disposal of these substances has led to the development of different regulation, establishment of Consortiums devoted to WCOs collection, even if to date they are mandatory only for industrial activities, while management of WCOs produced by citizens, defined as “urban waste”, is in charge of municipalities.

6. WCOs Composition and Pre-Treatments

In general, WCOs are mainly composed of 95% triglycerides with aliphatic chains ranging from 16 to 18 carbon atoms [101], deriving from palm, soybean, canola, sunflower, peanut, cottonseed, coconut, olive, and corn oils [3] (Figure 1a).

WCOs are mostly produced from food frying at temperatures between 150 °C and 200 °C and cannot be reused for food processing [54,102,103]. In fact, during use, vegetable oils undergo three main chemical reactions, i.e. hydrolysis, oxidation, and polymerization which alter their physical chemical characteristics [3,47,101] (Figure 3). Hydrolysis occurs due to the presence of moisture in food, forming glycerol, and Free Fatty Acids (FFA), leading to changes in color and forming smoke. Oxidation reactions occur as a natural reaction between double bonds present in vegetable oil and atmospheric oxygen, forming highly reactive hydroperoxides. Polymerization reactions may occur between unsaturated fatty acyl groups, forming dimeric or oligomeric triacyl glycerides [47,54]. Additionally, thermal cracking of unsaturated fatty acids, followed by oxidation at high temperatures may also promote polymerization reactions, originating a variety of toxic compounds such as heterocyclics [104].

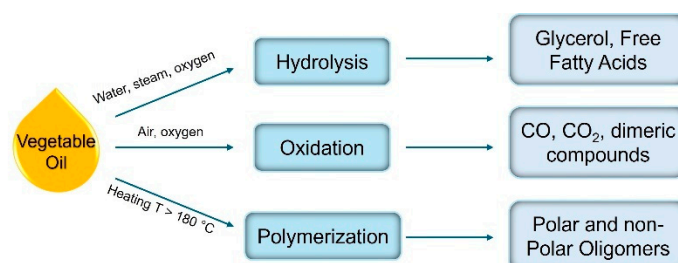


Figure 3. Chemical degradation reactions of vegetable oil.

Because of chemical degradation [25,27] of WCOs differ in color, viscosity, density, pH, flash point, number of unsaturated bonds, chemical composition, toxic components [13,58,105–107]. Data reported in the literature clearly highlight changes in pH (from 5.34 to 7.38), kinematic viscosity (from 28.744 mm²/s to 68.568 mm²/s) and molecular weight of WCOs generated by different vegetable oils or different cooking conditions (Table 1) [107,108] and for this reason many different pre-treatment methodologies have been reported in the literature for WCOs prior to recycling, such as filtration, extraction and distillation to eliminate water and impurities [26,109].

Additionally, WCOs recycling depends on its acidic, iodine value, number of conjugated diene or trienes making their recyclability more complex [54]. For example, it is evident that highly acidic WCOs will need to be neutralized if base-catalyzed transesterification has to be performed [27]. Further pre-treatments may be necessary to meet specific standards such as for example EN 14214 for biodiesel [110], while specific standards related to the volatile components present in WCOs must be met for polymers synthesis [27,54].

A very interesting work has been published by Mannu and co-workers reporting an extensive characterization of different WCOs subjected to several different frying cycles and purified with different methods (pH, Temperature) [45]. This complex study was carried out in order to increase

the standardization of pre-treatment procedures and improve recyclability of different WCOs. In fact, according to the authors, the combination of data acquired allowed to determine the best recycling protocols for a given WCO [44,45].

Table 1. Chemical physical characteristics of vegetable oils and WCOs derived.

Component	Sunflower oil ^(a)	Sunflower oil WCO ^(b)	Rapeseed oil ^(a)	Rapeseed oil WCO ^(a)	Palm Oil ^(a)	Palm oil WCO ^(a)	Sun foil ^(a)	Sun foil WCO ^(a)
Saturated Fatty Acids	71.5	32.0				80.0-93.0	74.4	73-15
Monounsaturated Fatty Acids	-	62.0				20.0-7.0		27-6
Polyunsaturated Fatty Acids	28.5	6.0					25.6	0-79
Acidic value (mg KOH/g oil)	0.30	2.29	0.06	1.06		0.66-1.13		0.72-1.44
pH	7.38	5.34			6.34	5.73-6.19	8.63	6.14-6.61
Density at 20°C (kg/m³)	919.21	920.40	918.00	929.00	919.48	923.2-913.4	919.6	919.8-923.2
Kinetic Viscosity at 40°C (mm²/s)	28.744	31.381	63.286	68.568	27.962	44.254-38.407	28.224	43.521-35.236
Molecular weight (g/mol)	670.82	51.94	869.16	871.01	535.08	135.66-586.05	119.71	55.18-395.28

^a Ref. 13. ^b Ref. 108.

Leaving aside its main use for biodiesel production, WCOs can be chemically modified and used as bio lubricants [47,111], bio surfactants [107,112], bio-based plasticizers [113–115], for the production of polymers and polymer additives [54,56,116,117], binders and additive for road and constructions [50,118–121], bio solvents [31]. All these applications will be further discussed in separate sections.

7. WCOs for Bio Lubricants and Bio Surfactants

7.1. Bio Lubricants

Bio lubricants (BL) are anti-friction agents with improved viscosity, higher thermal tolerance, lower volatility, compared with petroleum-based lubricants. BL world market was of 2 Mio\$ in 2022 and is projected to reach a value of 2.8 Mio\$ by 2031, registering a CAGR of 3.8% during the forecast period 2023-2031 [122]. WCOs are an efficient and environmentally sustainable feedstock for BL due to their high lubricity, biodegradability, low volatility and cost. Different modification routes have been reported for the conversion of WCOs into BL such as hydrolysis, esterification, transesterification, hydrogenation and epoxidation using both chemical and enzymatic catalyzed reactions [111,123–127] summarized in Figure 4.

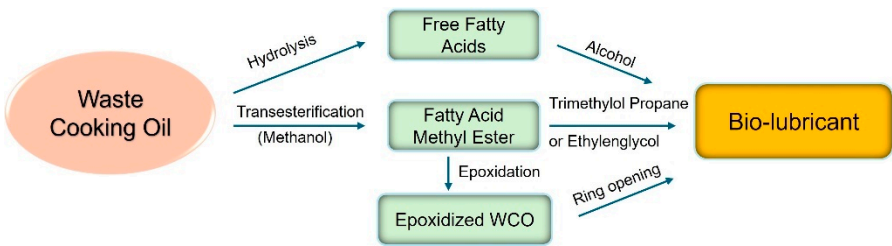


Figure 4. Main chemical reactions for the production of bio lubricants from WCO.

The different routes foresee hydrolysis of WCOs to FFAs followed by esterification with high molecular weight alcohols or polyols [128–130], or transesterification of WCOs to FAME followed by transesterification with high molecular weight alcohols such as trimethylol propane (TMP) [131–136],

or other polyols [111,137]. Alternatively, FAME may be epoxidized with hydrogen peroxide and acetic acid [128,138,139], followed by epoxy ring opening by alcoholysis or hydrolysis in the presence of a catalyst [133;135,140,141].

From an industrial point of view transesterification is one of the most feasible routes for the conversion of WCOs into BL and consequently the most investigated [111,135,142–144]. Although in most cases high conversions are reported, nevertheless BL produced by transesterification reaction have low viscosity index and modest performances at low temperatures.

Zhang et al. improved the efficiency of the process [127] employing lipase and [HMIIm][PF₆], a recyclable ionic liquid used as catalyst, giving a product with excellent lubricant properties, such as low pour point (-61 °C), high viscosity index (149) and high thermal-oxidation stability (312 °C). Sarno and coworkers adopted a similar transesterification process starting with WCOs and neopentyl glycol for the production of bio-lubricants [145]. A lipase immobilized on nanoparticles of modified magnetite was employed as catalyst showing high activity and recyclability up to ten times.

Alternatively, Jahromi and coworkers also explored the improved thermophysical and rheological properties of BL generated by epoxidation in the presence of cyclic oxygenated compounds (cyclopentanone, cyclopentanol, anisole, and 2-methylfuran) via a four-step pathway: hydrolysis, dehydration/ketonization, Friedel–Crafts acylation/alkylation, and hydrotreatment [140]. The process allowed to achieve BLs with low pour-point (-12 °C), kinematic viscosity of 47.5 cP (at 40 °C), viscosity index 186, and Noack volatility of 17 wt %, comparable to commercially available lubricants.

7.2. Bio Surfactants

Another interesting application of WCOs is for the production of bio-surfactants (BS), employed by the food, cosmetic, textile, health, pharmaceutical, mining and paper industry [100,112,146–152]. The market of BS has grown constantly during the last two decades and is expected to reach 4.8 Mio€ by 2025 [153].

BS are secondary metabolites generated by a variety of microorganisms such as fungi (yeasts) or bacteria (actinomycetes), that can either remain attached to the cell surface or be secreted outside the cell [154,155]. As for BL, also BS have several advantages over their synthetic equivalents since they can be produced from renewable feedstocks by fermentation, are environmentally compatible, and show better foaming properties in a wide range of conditions (pH, salinity, and temperature) [34,156].

BS have been classified either by their molecular weight (high and low molecular weight), ionic charges (anionic, cationic, non-ionic and neutral) and secretion type (intracellular, extracellular and adhered to microbial cells). However, their identification based on chemical structure remains the most widely adopted, divided in glycolipids, lipopeptides, fatty acids, phospholipids, neutral lipids and polymeric surfactants [149]. Glycolipids are low-molecular-weight amphiphiles that consist of a hydrophilic polysaccharide headgroup and one or more hydrophobic fatty acid tails [153,157], which may be produced from WCOs [158–160]. In fact, WCOs are an interesting feedstock for BS which may be converted by various yeast species in sophorolipids, rhamnolipids, mannosylerythritol lipids, and other glycolipids [107,158,159]. Several microbes such as *Bacillus*, *Pseudomonas*, *Acinetobacter*, and *Candida* have been extensively reported for biosurfactant production with different methodologies (metagenomics, metatranscriptomics, and metaproteomics) [158]. Nevertheless, to date BS are not widely employed, due to unsustainable production costs [100].

Chemical modification of WCOs for BS production could be a more economically sustainable alternative and therefore has been extensively studied. For example, Yusuff and coworkers recently reported the use of WCOs as a low-cost carbon source to produce anionic surfactant fatty acid methyl ester sulfonate via a transesterification-sulfonation process [161]. Furthermore, Permadani developed a bio-detergent from methyl ester sulfonated (MES) WCOs using titanium dioxide nanoparticles as catalyst [162]. In fact, several studies have been reported in the literature on the production of MES from WCOs [100,163] and are among the leading renewable surfactants and detergents at industrial scale, exhibiting superior detergency efficiency compared to fossil-based products, lower toxicity and

better skin compatibility [105,162]. The production of MES from WCOs foresees preliminary purification, followed by transesterification to form the corresponding methyl esters which are then reacted with sodium bisulfite as sulfonating agent [105]. Various papers dealing with the environmental performance of BS using WCOs as feedstock clearly demonstrate that their use significantly reduces environmental impacts compared to the use of other feedstocks [158,159,164].

In the field of branched non-ionic surfactants, Dong and coworker synthesized a new series of bio-based branched non-ionic surfactants (ethoxylated dihydroxy stearic acid methyl ester, DMOE), from renewable oleic acid derived from WCOs [165]. Results show that DMOE, has low foaming, strong defoaming ability, good wetting performance and outstanding emulsifying capabilities enabling the application of these surfactants for industrial cleaning activities.

Another interesting study has been published by Khalaf and coworkers [166] on the preparation and use of cationic gemini surfactants prepared from WCOs, as green inhibitors against acidic corrosion of N80-steel. Gemini surfactants are highly demanded worldwide also for their promising applications in the paper, medical hygiene, and textile industry [167]. Additionally, cost-effective and efficient adsorbent macro-porous organic polymers based on WCOs were synthesized on Kgs scale by Wu and co-workers, allowing to remove oil and heavy metals from wastewater ($\geq 94\%$), exhibiting excellent hydrophobic and super oleophilic properties [119].

8. WCOs for Polymer Additives

With the introduction of new regulations that are increasingly restrictive in the use of components classified as toxic, research is opening new horizons for the use of new bio based components as additives in the plastics industry [31,113]. The addition of plasticizers in polymers formulation plays a very important role in modifying the chemical and physical properties of plastic manufacts [31,168,169]. Plasticizers have been extensively used to produce flexible polyvinyl chloride (PVC) plastics, which otherwise are hard and brittle [170–172]. Phthalate esters, conventionally employed at industrial level for PVC production are known to exhibit a migration phenomenon from the polymer matrix to the surrounding media and to accumulate in the environment [113,168,172–174] and are subject to REACH restriction.

Due to their negative impact on human health and the environment, they have been banned in several countries for the production of toys and packaging materials [115,175,176]. In this context the syntheses of epoxidized soybean oil (ESO), methyl epoxy soyate, amyl epoxy soyate, acetylated derivatives of castor oil, tall-oil fatty esters, dicapryl sebacate have been described as alternative ecofriendly substitutes of phthalate esters, yet with limited applications due to high costs of starting materials used [173,177,178]. More sustainable alternatives have recently been reported employing WCOs as feedstock [114,115,176,179–182].

As reported by Liu et al. [180], acetylated-fatty acid methyl ester-malic acid ester (AC-FAME-MAE), obtained by modification of WCOs (Figure 5, route I), may be used as additive in PVC formulations giving a product with chemical and physical performances in line with the common industrial additive (phthalate), making it an excellent environmentally safe substitute to phthalate esters. Cai and co-workers also reported the synthesis of bio-plasticizers from WCOs according to the strategy reported in Figure 5, route II and III [179]. Two different highly epoxidized compounds (EGE-WCO and EP-WCO) were obtained and their characteristics as plasticizers for PVC tested. EGE-WCO with an epoxy value of 6.57% showed a high compatibility with PVC and plasticizing efficiency, decreasing Tg of PVC films from 62.8°C to 15.8°C and improving elongation at break. Migration phenomena of EGE-WCO were found to be negligible.

Additionally, Jia and co-workers proposed the synthesis of a covalently bonded plasticizer to the PVC backbone (WCOs methyl esters), avoiding possible migration phenomena which have also been reported with some WCOs based plasticizers [113].

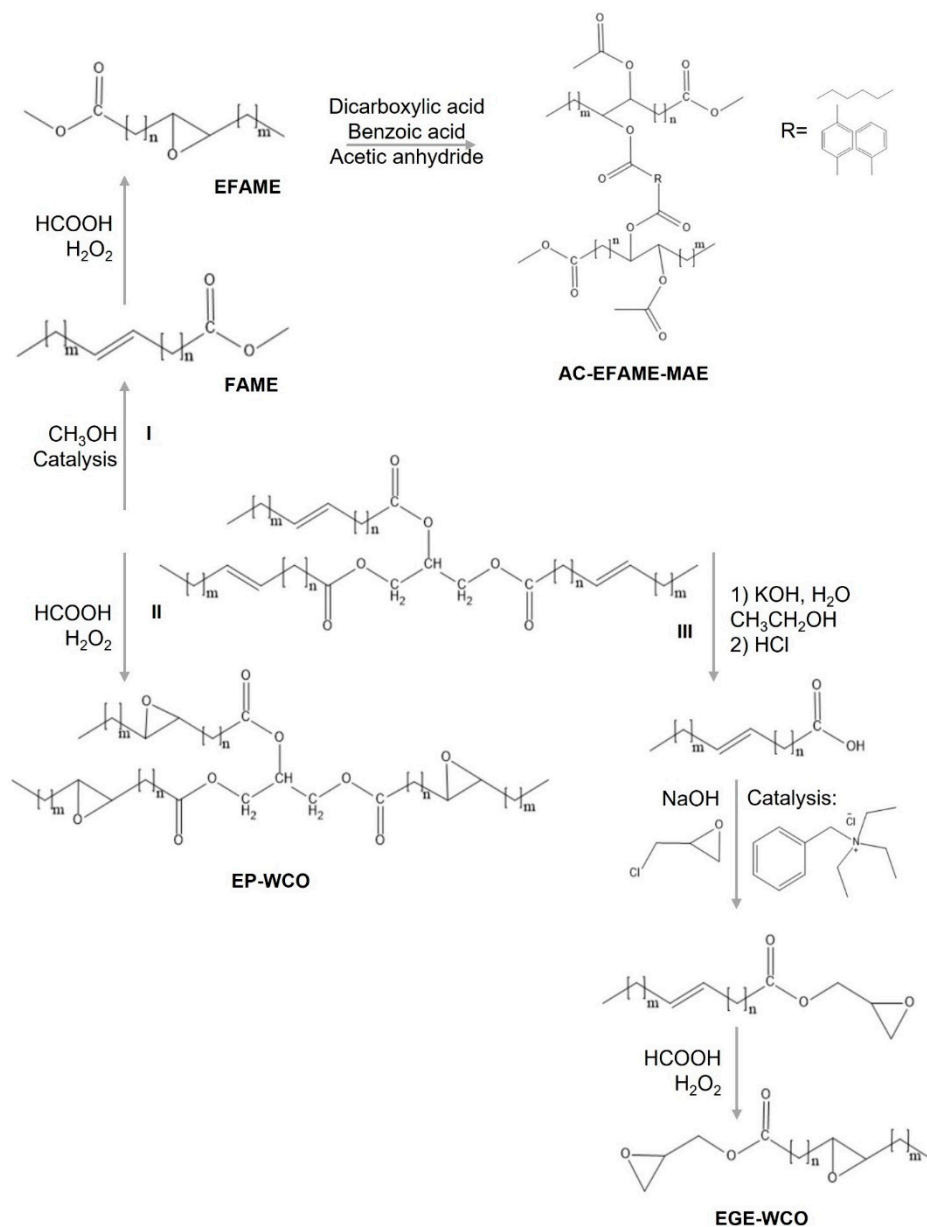


Figure 5. Examples of PVC additives prepared from WCOs.

9. WCOs for Polymer Synthesis

9.1. Polyurethane

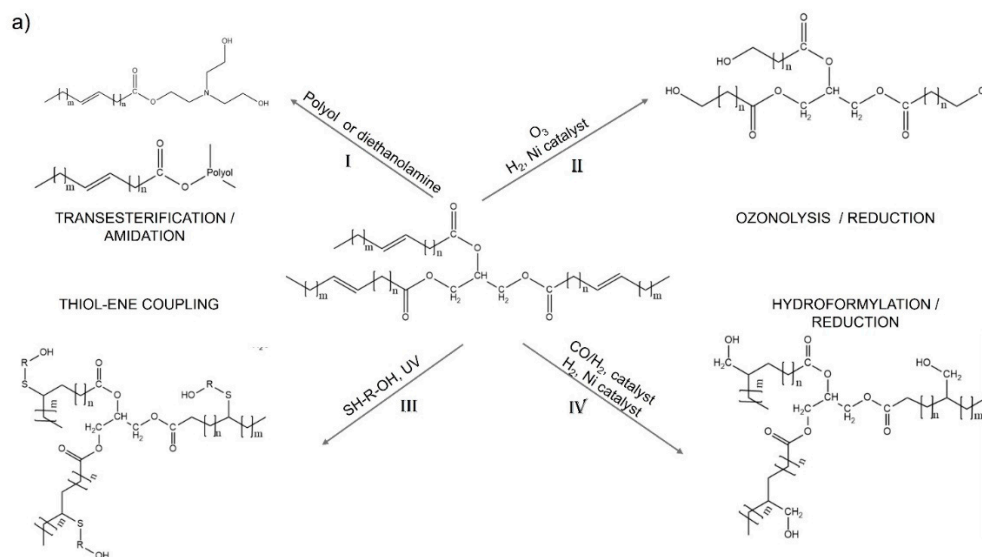
Polyurethanes (PUs) are an important class of polymers with a global market of about 26 Miot in 2022, estimated to reach 31 Miot by 2030, positioning PUs as a major player in the polymer market [44,183]. In fact, due to the broad chemical and physical characteristics of these polymers, it is possible to produce flexible and rigid foams, or nonporous materials for different applications ranging from the automotive sector to the construction industry [184,185]. Currently, PUs are prepared from virgin naphtha, however, scientific research is pushing towards replacement with renewable raw materials. In this context, many efforts have been made to prepare bio-based polyols from WCOs and achieve more renewable and sustainable PUs [56,186–189].

Although many strategies have been used for the production of polyols from vegetable oils, as for example transesterification, epoxidation, ozonolysis, thiolene coupling reaction, hydroformylation, and photochemical oxidation (Figure 6) [184,190–193], polyols from WCOs are

mainly produced by transesterification or epoxidation (Figure 6a, route I and Figure 6b) [183,188,194,195].

Polyols generated from the transesterification of WCOs are mixtures of triglycerides, monoglycerides and diglycerides together with glycerol (Figure 6a, route I). Since transesterification is an equilibrium reaction, to achieve high ester yields, an excess of alcohol, in most cases methanol, must be used to shift the equilibrium towards the products. A catalyst (alkali, acid or enzyme) is also added to improve the reaction rate, and esters yields [196–198].

Kuranska and coworkers reported the synthesis of polyols from WCOs in the presence of ethylene glycol, propylene glycol, diethylene glycol and triethanolamine (Figure 6a, route I), and their characterization by Gel Permeation Chromatography [195,199]. Taking into consideration the principles of Green Chemistry, the work by Kuranska and coworkers tried to maximize the incorporation of all reagents used in the final product, avoiding solvents and employing renewable reagents, so that bio polyols produced with optimized reaction conditions could be used for PUs synthesis without purification [199]. A different approach was reported by various researchers, who reported the modification of WCOs by epoxidation and ring-opening reactions, allowing to obtain rigid polyurethane foam with chemical and physical properties alike common polyurethane materials used in building and construction [194,200,201]. Particular attention has been given to the optimization of the catalysts employed for epoxidation and ring opening reactions and data reported demonstrate that Amberlite IR-120, an ion exchange resin, and tetrafluoro boric acid gave highest yields and selectivities [200,201]. Interestingly, it has also been verified that changing the additives used to prepare PUs containing WCOs polyols allowed to modulate physical-mechanical characteristics and biodegradability of PUs [201]. For example, combination of polyethylene glycol (PEG), 4,4'-diphenylmethane diisocyanate (MDI) and WCOs, allowed to achieve biodegradable PU sheets.



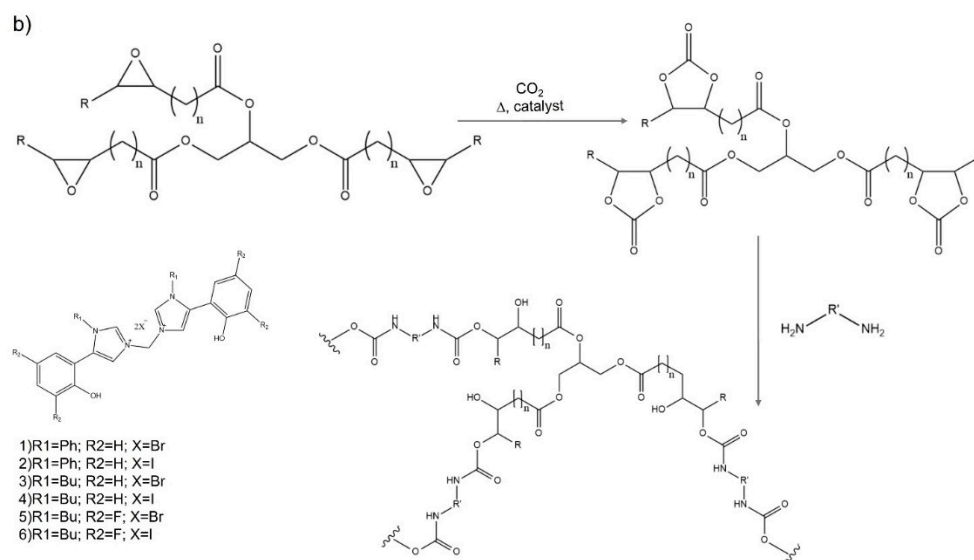


Figure 6. Different strategies for the synthesis of polyols from vegetable oils and WCOs.

PU prepared with WCOs having adequate properties for solid polymer electrolytes production was reported by Salleh and co-workers [188] by pre-polymerization (epoxidation and hydrolysis), in the presence of lithium iodide as charge carrier and ethylene carbonate. Finally, MDI was added and films casted [188,202,203]. Few other works have been published on the production of composite materials containing PUs prepared from WCOs derived polyols. For instance, Lubis and coworkers [204] tested the preparation of reinforced sugar palm fibers for PU foams preparation from WCOs, polyols and toluene diisocyanate. According to the authors WCOs promote cross-linking and improve the interfacial adhesion between the fiber and the matrix. Additionally, WCOs have been employed as precursors for super-hydrophobic coating applications through a process of amidation of WCOs, followed by functionalization with diisocyanates and fatty acids [205] or with diisocyanates and amino-terminated polydimethylsiloxane [179].

Silva and coworkers recently reported a comparative life cycle analysis on fossil and bio based polyurethane foams evidencing different benefits and criticalities encountered by PU production [206]. A major concern remains on the use of toxic diisocyanates employed also for the production of PUs with bio-based polyols. Alternatively, non-isocyanate polyurethanes (NIPU) have been widely studied, yet their industrial application is still hampered by low physical-chemical properties of PUs obtained, together with unsustainable production costs. Nevertheless, NIPU constitutes the most promising frontier to produce environmentally sustainable PUs and should therefore be implemented.

Within this panorama the efficient synthesis of polyurethanes from WCOs and CO₂, in the presence of different organocatalysts are a promising alternative (Figure 6b) [207,208]. These interesting works not only promote the recovery and reuse of a waste (WCOs), but also imply the consumption of CO₂, thus helping to reduce greenhouse emissions, avoiding the use of fossil-based chemicals and toxic diisocyanates. Werlinger and coworkers reported the synthesis of bis-imidazole salts catalysts (Figure 6b) bearing two -OH groups for the synthesis of cyclic carbonates derived from WCOs, employed as starting materials for the synthesis of different NIPUs by polyaddition reaction of carbonated vegetable oils with a variety of diamines. Catalysts and cyclic carbonates prepared were characterized by NMR, whereas NIPUs were characterized by NMR, IR, GPC, and their thermal properties studied by TGA and DSC analysis.

9.2. Acrylic Polymers

A wide range of applications exist for the use of vegetable oil triglycerides to prepare acrylic polymers, and similar protocols have also been adopted starting from WCOs [54,209].

For example, Wu and coworkers [210,211] reported that they successfully converted WCOs from McDonald restaurants into acrylic polymers via a straightforward one step reaction for additive 3D printing manufacturing. Filtered WCOs were reacted with acrylic acid in the presence of boron trifluoride etherate ($\text{BF}_3 \cdot \text{Et}_2\text{O}$) at 80°C for 4 h. The product was characterized, showing that the double bonds of the triglyceride molecule broke as a consequence of the grafting of the acrylic groups. After purification to eliminate residual unreacted acrylic acid, the product could be 3D printed with or without the addition of a photo inhibitor. Interestingly, the polymers were found to be biodegradable, showing 18% weight loss after 14 days incubation in soil [212]. Further papers have also studied 4D Yan Liu, printing of multifunctional photocurable resin based on waste cooking oil [135].

Onn and coworkers recently reported the modification of WCOs through enzymatic acidolysis carried out to increase unsaturation sites and chemical reactivity of WCOs to produce an acrylic pre-polymer which was photo crosslinked in the presence of a photo initiator [209].

9.3. Alkyd Esters

Alkyd ester resins are prepared by condensation reaction between a polyol and polybasic acid and a fatty acid. Alkyd esters are used in paint, varnishes, and casting molds with a yearly world production of around 200,000 tons [213]. Fatty acids employed for alkyd resins formulation may derive from vegetable oils, animal fats or WCOs, improving their sustainability. A common strategy employed to produce alkyd resins from vegetable oils, known as the monoglyceride process, is reported in Figure 7 [54] and foresees a twostep strategy: i) base or acid catalyst alcoholysis of WCOs to generate monoglycerides and diglycerides; ii) polycondensation reaction of the monoglycerides with an anhydride (aromatic or aliphatic). According to the type and concentration of anhydride used, as well as the length of the oil the properties of alkyd ester resins can be changed.

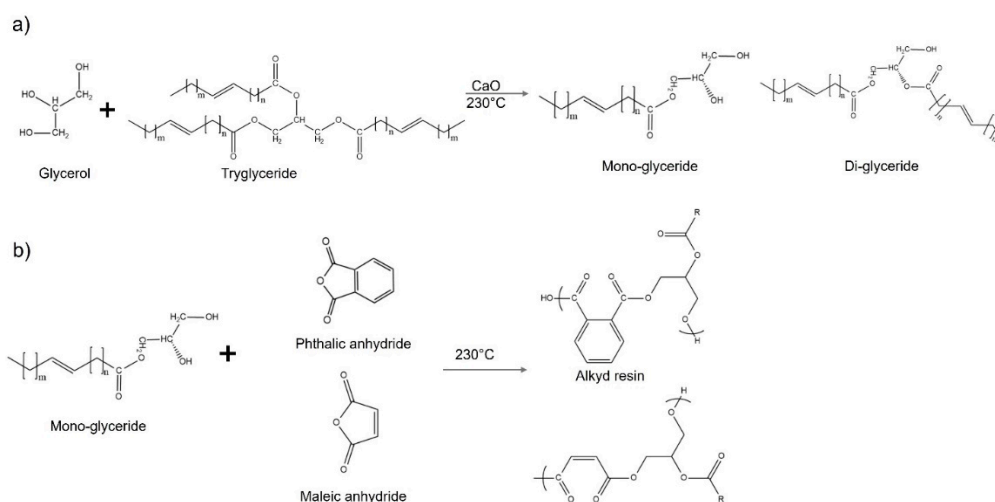


Figure 7. Monoglyceride process for the synthesis of alkyd resins.

Research carried out by Silvianti and coworkers [123] allowed to successfully synthesize alkyd ester resins from Waste Palm Cooking Oil using zeolites as water adsorbents in alternative to distillation and at the same time reducing the concentration of FFA content, contaminants, toxic products, bad odors and color. WCOs have also been employed to produce alkyd-based printing inks for vehicles by Phunphoem and co-workers (Phunphoem et al., 2020).

9.4. Epoxy Resins

Epoxy resins are thermosetting polymers made of monomers that have a three-membered epoxide ring. Epoxy resins are widely used for many applications thanks to their outstanding thermal and mechanical properties and worldwide consumption of epoxy thermo-set resins is expected to

reach 17.0 Mio\$ by 2028 [214]. The epoxy resin industry is presently based on diglycidyl ether of bisphenol A (DGEBA), produced by reaction between bisphenol A and epichlorohydrin. All these chemicals are listed as highly toxic for human health and DGEBA is identified as endocrine disruptor, teratogen and has long-term effects on aquatic life, and has therefore been banned from materials used in baby milk bottles [215]. Bio epoxy resins thus appear as a very important biobased, renewable, biodegradable and biocompatible alternative to petroleum-based epoxy resins and triglycerides coming from WCOs have been demonstrated to be applicable for the scope [216,217]. Epoxide resins are produced either by chain-growth ring-opening polymerization or step-growth polymerization [217]. Fernandes and co-workers are claimed to be the first to have used epoxy resins from WCOs in combination with recycled carbon fibres followed by casting to produce composites with interesting tensile strength (53 MPa), Young's Modulus (3.2 GPa) and reducing characteristic brittleness of DGEBA [218].

10. Waste Cooking Oil for Roads and Constructions

Increasing need of renewable resources as alternatives to fossil-based ones has pushed many researchers to study the use of WCOs also in the construction industry. In particular, due to its similar elemental composition to petroleum, WCOs have been widely employed for construction materials mainly for asphalt and concrete production as described below [134,211,219–227].

10.1. WCO for Asphalt Production

Many different works have been published in the last decade on the use of WCOs as additives for asphalt concrete and asphalt cement to improve various characteristics such as softening point, viscosity, complex modulus, creep stiffness, resistance to deformation, voids in mineral aggregate and elastic recovery [69,181,228–230]. On the other hand, in most cases WCOs additives adversely affect penetration, ductility, phase angle, stress relaxation, thermal cracking resistance and reduce overall stability of asphalt concrete [221,231,232].

Wang and coworkers investigated the use of WCOs as modifiers for fossil-based asphalt binders. Based on infrared spectroscopy, frequency sweep rheological analysis, multiple stress creep recovery test, and linear amplitude sweep tests, the authors verified that WCOs modified binders displayed higher carbonyl index, lower binder stiffness and softening effect and reduced rutting resistance at high temperatures [51]. Also fatigue life of bio binders under cyclic fatigue loading was significantly improved at increasing WCOs wt% by wt of bitumen, further confirming their potential applicability as sustainable asphalt binders [51].

Additionally, WCOs have been extensively studied for the recycling of Reclaimed Asphalt Pavement (RAP) and grinded tire rubber (GTR), that would otherwise be discarded [97,227,233].

The recycling of RAP generates economic savings, reducing the exploitation of non-renewable resources, energy consumption and polluting emissions in mining and transportation operations [234,235]. However, as bitumen ages, it loses its original properties causing distresses in pavement that may endanger traffic safety and reduce travel comfort. In fact, when quantities above 20 wt.% of RAP by wt. of bitumen are used, a gradual decrease of pavement's fatigue cracking, and low temperature cracking [236]. WCOs are an economically and environmentally sustainable alternative to fossil based rejuvenating agents allowing to solve these shortcomings [233]. Independently from the geographical origin of RAP and WCOs derivation, data reported in the literature generally agree that WCOs between 1-8% wt.% by weight of bitumen, positively affect penetration value [231,236–241]. This can be attributed to the alteration in the consistency of asphalt, contributing to improve fluidity and workability of the modified asphalt.

Pretreatment of WCOs is normally required to reduce acidity, due to variable quantities of FFAs present which negatively influences performances [50]. Common practices are esterification, transesterification in the presence of methanol or refining processes which promote the overall performance of WCOs modified bitumen [242–244]. An interesting study has recently been published by Bardella and coworkers on the possibility to achieve highly performing rejuvenating agents by

chemical modification of WCOs or hydrolysed WCOs by transesterification (other than methanol) or amidation reactions to achieve various WCOs esters and amides. All samples were characterised by nuclear magnetic resonance, melting, and boiling point. Efficiency of WCOs esters was assessed by means of the Asphaltenes Dispersant Test and the Heithaus Parameter showing that bitumen blends containing 25 wt.% of WCOs by weight of bitumen, modified with 2-phenylethyl alcohol, performed better than bitumen alone [50].

Alternatively, in his recent work, Enfrin and coworkers studied the pre-treatment of WCOs by epoxidation reaction, which were used as rejuvenating agents for RAP [245]. This low-cost procedure allowed to produce rejuvenating agents with immediate cracking resistance comparable to commercially available fossil based rejuvenating agents, and higher long-term cracking resistance. The authors suppose that epoxide groups promote dispersion of the asphaltene clusters of bitumen, creating bridges between the molecules and preventing their agglomeration, slowing ageing phenomena.

Another interesting work has been reported on the rejuvenating and self-healing efficiency of WCOs capsules preparation by mixing WCOs and sodium alginate or chitosan powder which was then poured in a CaCl_2 solution, and capsules produced with a micro peristaltic pump. In general, WCOs performances were superior to commercially available products and in particular fatigue healing performances of asphalt mixtures were improved by 10~30% [220].

From an environmental point of view, WCOs rejuvenating agents reduce greenhouse gas emissions and energy consumption. For instance, emissions of CO_2 are reduced from 20.06 to 18.44 $\text{KgCO}_2\text{e/t}$, switching from conventional bitumen to bio-bitumen and energy consumption respectively from 48.33×10^9 to 42.81×10^9 J for ton of material produced [127,246].

Elahi and coworkers very recently reported a study on the modification of WCOs with styrene-butadiene-styrene (SBS) from GTR to give a rubbery-solid used as bitumen additive. WCOs promote the swelling and cross-linking process of SBS, improving the adhesion between the modified WCOs and the bitumen [235]. A three-step process was used for the modification of WCOs foreseeing: i) filtration; ii) pre-swelling of SBS with various wt./wt.% of WCOs/SBS (45:55 and 50:50 wt./wt.%) at 160 °C; iii) modified WCOs mixtures were reduced in size and mixed with the bitumen (between 5 wt.% and 25 wt.% by weight of bitumen). Data reported show that increasing the content of SBS modified WCOs improve bitumen stiffening, temperature susceptibility, viscoelastic response and elasticity of the bitumen. Also, ethylene-vinyl acetate (EVA) has been used in combination with WCOs giving highly performing rejuvenating agents [53,246].

10.2. WCOs for Concrete

WCOs have also been studied for the production of sustainable cements. Li and co-workers developed a cement clinker and gypsum mixture containing different WCOs, and verified their physical mechanical characteristics, together with economic and environmental impacts [139]. Results show that, overall, WCOs favourably improved cement grinding and when highly unsaturated WCOs were employed also cement strength was improved. Additionally, WCOs improved microstructure density, hardening the cement paste. The authors concluded that the use of WCOs as grinding promoter in cement processing is economically and environmentally beneficial.

Recently Liu and co-workers studied the use of WCOs emulsions as shrinkage reducing admixture to improve concrete durability. Oil and water emulsions prepared were able to reduce cement shrinkage, enhance its dispersion and stability [247]. The addition of different contents of WCOs and water emulsions reduce the total porosity and refine the pore size by reducing significantly $\text{Ca}(\text{OH})_2$ crystals, probably as a consequence of saponification reaction between WCOs and $\text{Ca}(\text{OH})_2$.

11. WCOs as Bio Solvents

Another interesting characteristic of WCOs is their ability to capture volatile organic compounds (VOC) [31,59], originated from different manufacturing procedures such as emissions from rice husk pyrolysis, rubber vulcanization [59,248].

Lhuissier and coworkers [59] developed a non-aqueous-phase absorption and degradation bioreactor for Volatile Organic Compounds (VOC). VOC targeted were n-heptane, ethyl acetate, isopropanol, methyl isobutyl ketone, toluene, m-xylene and 1,3,5-trimethylbenzene. Results allowed to conclude that WCOs have an absorption efficiency which is comparable to fossil-based oils.

Tarnpradab and co-workers employed WCOs to reduce emissions produced during rice husk pyrolysis [248], and also in this case WCOs were able to reduce the content of organic hydrocarbons formed. Other different examples exist of the use of WCOs for the adsorption of VOC produced by the leather industry [249] or other industrial contaminants such as mercury [60].

In addition, WCOs have been employed as solvents for the pretreatment of GTR to compensate for the insufficient storage stability of GTR modified asphalt [221,250]. GTR is one of the most common bitumen modifiers used to improve bitumen rheological properties. However, bitumen containing GTR generally has poor storage stability and workability, affecting the performance of the pavement [221,250]. Pretreatment of GTR with WCOs reduces polymer segregation phenomena, improving ageing resistance and reducing environmental impacts [251,252]. In addition, recent research also indicated that a rejuvenator with excellent storage stability and regeneration effects can be obtained when GTR is pyrolyzed in a WCOs rich phase [253].

12. Other Applications

Although different studies have recently reported the use of edible [254,255] and non-edible waste oils [256,257], as organic phase change materials, few literature data are available on the use of WCOs for the scope and applied to food preservation, asphalt or building thermoregulation [258–262].

Different works have been reported on the use of filtered WCOs for thermal energy storage analogously to other virgin or waste oils [257,259]. LCA data were reported showing that WCOs allowed to reduce significantly energy consumption.

Alternatively, phase change capsules (PCC) were prepared by mixing WCOs in the presence of sodium alginate and Tween80, an emulsifying agent, and microcapsules prepared by microfluidic technology [258]. PCC were employed for the preparation of foamed concrete having mechanical properties within standard requirements (strength >4.80 MPa), with interesting thermal insulation properties.

13. Conclusions

According to the literature it evidently emerges that WCOs are a valuable food waste that can be used as feedstock to produce a variety of different bio based polymeric materials. Although WCOs are renewable starting materials for a sustainable biorefinery, nonetheless they struggle to find industrial applications. Complexity and variability of WCOs composition together with an inadequate collecting system are among the main barriers for the introduction of WCOs as feedstock at industrial level. Competing use of WCOs for biofuel market is also a drawback, reducing its availability.

To overcome these problems different legislative strategies should be adopted in order to harmonize throughout EU the collecting system and promoting an awareness campaign among consumers on the importance of WCOs separate collection.

From a technological point of view many different strategies and applications exist for the production of high value polymeric products from WCOs although only few of them have been implemented at industrial level today. This evidently testifies that further efforts need to be done both by the scientific community and the industry to reduce the complexity of processes necessary to

transform WCOs in polymeric materials and is strictly connected also to the capacities of WCOs collecting systems and management.

Once WCOs will be collected, managed and used properly, they will surely become a valuable feedstock for the polymer industry satisfying most of the Sustainable development goals of the agenda 2030 and of the Principles of Green Chemistry.

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