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Reduction of Epoxy Resin VOCs Using Higher Fatty Acids from the Agricultural Industry: A Review

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Abstract: A disadvantage of many commonly used impregnants and resins is their high toxicity, related to the presence of harmful aromatic hydrocarbons and volatile organic compounds (VOCs) in their composition. VOCs account for a relatively large approx. 30% portion in the synthetic resins industry. One idea for reducing or eliminating VOCs from the production of resins, paints is the use of high-quality intermediates and biodegradable raw materials. A perspective on novel approaches to protecting concrete surfaces was presented, involving a concept of using two types of higher fatty acids for this purpose: stearic acid (STA) and methyl esters (ME). Recent technological advancements have centered on vegetable oil feedstocks for industrial applications. This is due to their suitability for industrial production of agents, as they substitute non-renewable hydrocarbons. The cited tests confirm the hydrophobic nature of coatings formed using STA and ME on various materials. From the analysis of the literature, it appears that the study of anticorrosion coatings with biodegradable admixtures, i.e. higher fatty acids, should be developed because of their promising results in efficiency, reduction of toxic substances (VOCs) and their impact on the environment.

Introduction

According to Fortune Business Insights, the global resins market was valued at $556 billion in 2023 and is forecast to increase from $582.8 billion in 2024 to $859.27 billion in 2032, representing a compound annual growth rate (CAGR) of 5% over this period. The Asia region dominated the resins market with a 50.57% share in 2023. The resins market in the United States is also projected to grow significantly, reaching $125.21 billion by 2032. In 2023, the construction segment accounted for a significant portion of the resins market (Fortune Business Insights, 2024). In terms of resin applications, the paints and coatings segment dominated, holding 38.6% of the market share in 2023 (Fact. MR, 2024) (Fig. 1).

The growth of the resin market is driven by infrastructure development in developing countries, as well as the renovation of existing old buildings. Epoxy, acrylic and phenolic resins mainly used for paint, coatings, adhesives and sealants encompass a wide range of applications. Most petrochemical raw materials for resins are produced as by-products of oil and natural gas production. The use of petroleum-based products results in the release of harmful substances into the environment, causing severe contamination of soil, water, and air. This process destroys ecosystems, weakens natural biodegradation mechanisms, and, moreover, petroleum-derived substances exhibit mutagenic and carcinogenic effects (Vogt and Topolska 2023). Crude oil, a key feedstock, contains various volatile organic compounds (VOCs). Among gaseous air pollutants, a particularly significant group is odorants (odorous substances) - volatile compounds that are toxic, detectable at very low concentrations, and produce unpleasant odors (Dobrzyniewski et al. 2023). Long-term

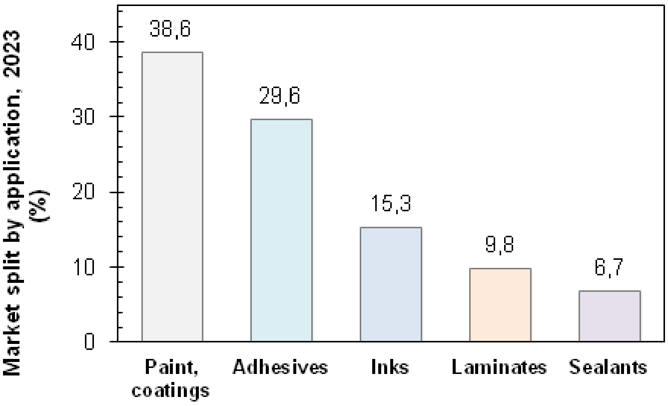


Fig. 1. Global resin solvents market in 2023 (own elaboration according to the Fact. MR, 2024).

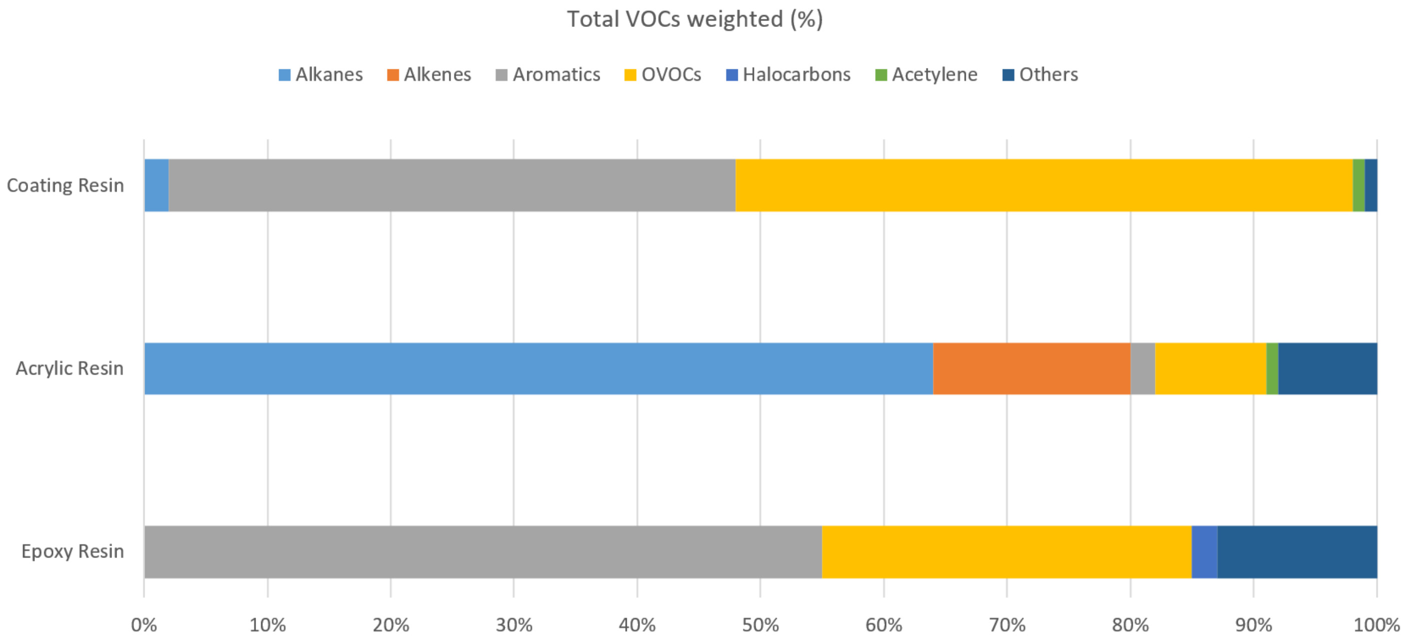
exposure to volatile organic compounds (VOCs) is common, as these substances are widely used in various manufacturing processes such as glue and pharmaceutical production, as well as in industrial solvents. VOCs are also commonly present in households, found in products like petroleum fuels, paints, and cleaning agents (Brochocka et al. 2021).

VOCs account for approximately 30% of the synthetic resins industry (Yao et al. 2021). They play a significant role in the formation of secondary pollutants, oxidation processes in the atmosphere, and negatively affect human health. VOCs can photochemically react with nitrogen oxides (NOx), leading to the formation of ozone, fine particulate matter (PM 2.5), secondary organic aerosols (SOA), and contributing to urban photochemical smog (Xiao et al. 2024a). Many impregnants and resins commonly used in the industry have several disadvantages, including high toxicity due to the presence of harmful aromatic hydrocarbons, flammability (Class II), unpleasant odors, and environmental aggressiveness (Gao et al. 2021). The current raw materials for epoxy resin production, such as bisphenol A and epichlorohydrin, primarily come from petrochemical sources. Thus, these agents often contain volatile organic compounds, which are chemicals that readily evaporate at room temperature, contributing significantly to air pollution and climate change.

Toluene, benzene, styrene, 1,4-dioxane, methylisobutylketone, dichloromethane and propane are among the most abundant substances found in synthetic resins. Ma et al. (Ma et al. 2021) studied the composition of VOCs in various synthetic resins. Three selected ones relevant to the subject of this article are presented in Figure 2.

In the synthetic resin industry, a significant quantity of aromatic raw materials and oxidized VOCs (known as OVOCs) are used. Epoxy resin consists of 55% aromatic hydrocarbons, while acrylic resin, an alkane resin, contains 64% alkanes (see Fig. 2) (Yao et al. 2021, Ma et al. 2021).

As reported by Ma et al., styrene is the main pollutant in synthetic resins. Epoxy and acrylic resins are considered medium ozone-generating resins. Between 2005 and 2018, emissions from synthetic resin production increased from 77.6 Gg to 287.7 Gg (Ma et al., 2021). According to Ma et al. the oil-based paint production is characterized by the highest reactivity. The ozone-producing potential of VOCs should not be overlooked, as the use of hydrocarbon solvents in high concentrations can pose health risks. VOCs can cause allergies and cancer in humans, and long-term inhalation of VOCs is very dangerous to the respiratory system (Parker et al. 2013). In particular, benzene, toluene, xylene, ethylbenzene, and formaldehyde are recognized carcinogens (Wang et al. 2024). According to Wang et al., toluene emitted during painting is neurotoxic to the reproductive system, causes DNA damage in the developing brain, and leads to liver damage (Wang et al. 2017). The authors report that painters' exposure to inhaled VOCs during painting jobs ranged from 3.5×10^3 to 14.8×10^3 mg/ m^3 for solvent-based paints. For instance, ethylbenzene and 1,2-dichloropropane used in the coating industry have been linked to increased cancer risks.



	Epoxy Resin	Acrylic Resin	Coating Resin
		content in percent	
Alkanes		64	2
Alkenes		16	
Aromatics	55	2	46
OVOCs	30	9	50
Halocarbons	2		
Acetylene		1	1
Others	13	8	1

Fig. 2. Percentage VOC compositions of the epoxy, acrylic and coating resins (own elaboration based on Ma et al. 2021).

The issue of VOC emissions has become one of the key challenges facing today's chemical industry. On April 21, 2004, the European Parliament issued Directive 2004/42/EC, which limits the emissions of volatile organic compounds due to the use of organic solvents in certain paints, varnishes, and vehicle refinishing products. The measure was introduced because VOCs play a significant role in the local and transboundary production of photochemical oxidants within the troposphere's boundary layer. Since 2007, the European Union (EU) Solvent Emissions Directive (SED) 1999/13/EC has required all installations producing more than 5 tons of VOCs annually to adopt a solvent reduction approach (Directive 1999/13/EC of March 11, 1999).

Efforts are being made to develop new agents with favorable technical characteristics that also consider ecological aspects. One approach to reducing or eliminating VOCs in the production of resins, paints, and impregnants is the use of high-quality intermediates and biodegradable raw materials. In recent years, consumer demands, regulations and an emphasis on sustainability have driven increased demand for green raw materials. As a result, there has seen growing interest in high-solids resins and water-based resins, which significantly reduce VOC emissions. Rather than relying on highly toxic, chemical-based agents, companies are increasingly turning to innovative formulations with reduced VOC content, such as water-soluble impregnations (Hodul et al. 2024). While solvent-based technology releases 65% of VOCs, water-based coatings release only 5% of their VOC content, and in many cases, even less.

The global market for water-based resins was valued at \$52.6 billion in 2023 and is projected to reach \$84.85 billion by 2032. In 2023, the Asia-Pacific region dominated the market, accounting for 42.2% of the market share (Vantage Market Research, 2023). The global market for water-soluble polymers was estimated at \$38.56 billion in 2023, and it is expected to increase from \$40.66 billion in 2024 to \$62.96 billion by 2032 (Fortune Business Insights, 2024).

Replacing solvent-based coatings with their water-based alternatives in the automotive industry has helped reduce VOC emissions per car from 756.5 to 489.6 grams (Xiao et al. 2024b).

The growth of the green economy, increased awareness of carbon footprints, and emphasis on sustainable systems and life-cycle analysis are encouraging many thermoset resin manufacturers to shift from petroleum-based raw materials and to agricultural alternatives.

Reducing VOC emissions is an important aspect of sustainable development strategies, as it directly impacts air quality and human health. Sustainable development emphasizes the reduction of VOC emissions across various sectors, including the chemical, automotive and construction industries. This is essential for reducing air pollution, improving air quality, and safeguarding human health (Qian et al. 2024). The adoption of low VOC-emitting technologies, such as paints, adhesives, waterproofing agents, and cleaners, supports this goal. Consequently, there has been a strong trend in industrialized countries with advanced technical capabilities and environmentally conscious societies to develop resins that are both harmless and environmentally friendly. Some conventional polymers, such as polyurethane, polyester and epoxy, can now be synthesized from bio-based materials, including vegetable oils and cellulose (Ramezanpour et al. 2024).

Recent technological advancements have focused on utilizing vegetable oil feedstocks for industrial applications. These feedstocks are particularly suitable for producing industrial agents, as they can substitute non-renewable hydrocarbons. Compared to petroleum, bio-oils offer significant advantages: they are more accessible, renewable, biodegradable, possess a higher flash point, and contain fewer aromatic compounds.

Recent studies suggest the potential use of higher fatty acids for hydrophobizing concrete exposed to corrosive environments (Barnat-Hunek et al. 2023). Owing to their hydrophobic properties, these acids could be applied in the production of mold release agents and corrosion inhibitors.

According to the literature, the contact angle of materials serves as an indicator of their wettability. High wettability (hydrophilicity) is characterized by a small contact angle ($\theta < 90^\circ$), while low wettability (hydrophobicity) is associated with a large contact angle ($\theta > 90^\circ$). A droplet is most often spherical in shape, with its height denoted as h and the radius of the contact surface as r. The wetting angle θ is the angle formed between the surface of the solid and the tangent to the droplet surface, drawn from the point of contact of the three phases – solid, liquid, and air (Fig. 3) (Rudawska 2013).

The primary chemical characteristic of vegetable oils, which are predominantly fatty acid triglycerides, influencing their potential for direct use as lubricants is the degree of unsaturation in fatty acids. This characteristic can vary greatly depending on the origin and growing conditions of the plants. From an environmental perspective, the most favorable functional property of vegetable oils is their high biodegradability, allowing them to naturally degrade into CO_2 and H_2O (Fiszer and Szałajko 2000). Compared to petroleum oils and even synthetic esters, vegetable oils show the highest biodegradability - in the range of 80-100% - regardless of

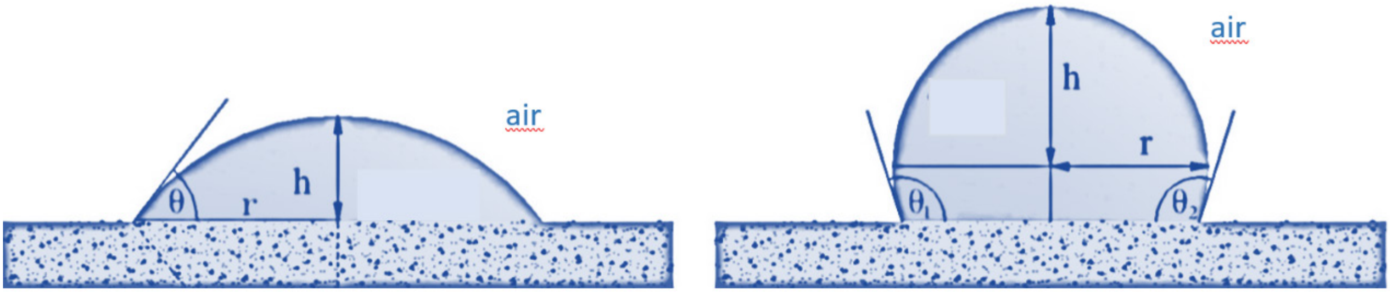


Fig. 3. The contact angle of a solid with a liquid (own elaboration based on Rudawska 2013)

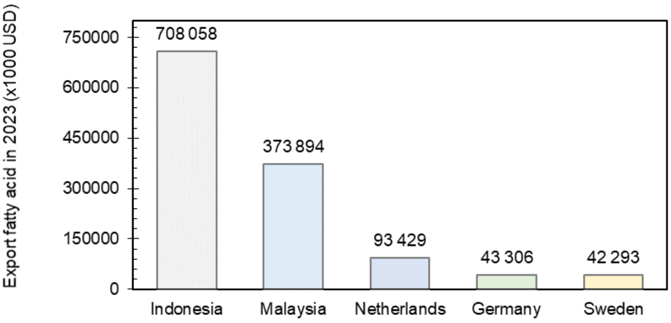


Fig. 4. Export fatty acid, acid oil in 2023 by world's largest exporters (compiled from World Bank, 2024).

origin, and are classified with a harm-to-water degree of 0 on a four-step scale from 0 to 3) (Zajezierska 2016). Medium-chain triglycerides are expected to dominate the global fatty acid methyl esters (FAMEs) market due to growing demand. FAMEs are a category of fatty acid esters produced via the transesterification of fats with methanol. A byproduct of this process is methyl ester residue (MER), which originates from manufacturing plants. The global fatty acid methyl ester market was valued at $8,584 million in 2020, increased to $17,460 million in 2023, and is projected to reach $23,200 million and 2024, and $33,400 million by 2033 (Valuates Reports, 2024). In 2023, the largest exporters of monocarboxylic fatty acids and acid oils were Indonesia, Malaysia, the Netherlands, Germany, and Sweden (Fig. 4) (World Bank, 2024).

Fatty acids are highly appealing due to their non-fossil fuel origins, lower flammability compared to paraffins, and, in some cases, their ability to forgo microencapsulation (Yuan et al. 2014). Based on the type of methyl ester, the market is classified into palm oil methyl ester, soybean methyl ester, rapeseed methyl ester, and others (Fig. 5). Among these, rapeseed methyl ester dominated the market in 2022, holding a 40% market share (Custom Market Insights, 2024). As a critical component, rapeseed methyl ester plays a significant role in the renewable energy sector by serving as a sustainable alternative to traditional fossil fuels.

The authors concluded that the effect of anti-corrosion coatings with biodegradable additives on concrete durability has not been extensively studied. However, this area requires thorough preparation and a comprehensive research program, which will be addressed in their upcoming article. In this paper, the analysis focused on two types of higher fatty acids: stearic acid and methyl esters.

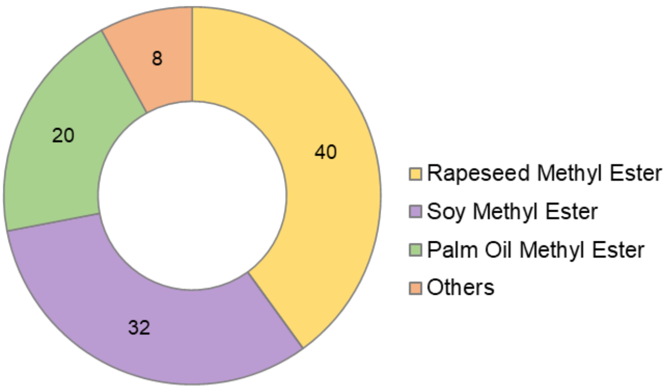


Fig. 5. Global fatty acid methyl esters market 2024-2033 (%) (by type) (own elaboration based on Custom Market Insights, 2024)

General principles and methods of surface protection

The PN-EN 1504-2:2006 standard outlines surface protection methods for reinforced concrete structures. It identifies material and structural protection as key approaches to ensure the durability of a structure. These methods include using concrete with increased integrity (based on exposure classes defined in PN-EN 206+A1), employing products and systems that strengthen the material's structure, such as admixtures and additives, as well as the use of cements and aggregates with greater resistance to aggressive chemical environments. While structural protection is widely implemented, the surface protection of structural components is an equally popular solution. EN 1504-2 specifies three types of corrosion protection: hydrophobic impregnation, impregnation, and coating.

Each of these protection methods, in addition to protecting against staining, also affects the durability of concrete elements by:

- protecting against harmful agents such as water or other liquids, vapors, gases, or chemical and biological agents (principle 1 (PI)),
- regulating moisture by adjusting and maintaining moisture in the concrete at a preset level (principle 2 (MC)),
- increasing resistance to physical agents (principle 5 (PR)),
- improving resistance to chemical agents (principle 6 (RC)).

Hydrophobizing impregnation – this technique involves the introduction of hydrophobic agents to the near-surface zone of the material, with the preparation penetrating several millimeters into the structure of the material. This method imparts water-repellent properties to the surface while leaving the capillaries and pores unfilled. Only the inner surfaces of the pores and capillaries are coated, transforming the material from hydrophilic to hydrophobic without altering its appearance. Additionally, some additives significantly reduce absorbability while maintaining high levels of water vapor diffusion.

For outdoor elements, weather conditions gradually degrade the coating, potentially leading to its complete disappearance. Organosilicone agents, such as silanes and polysiloxanes, are the most commonly used materials for these applications (Barnat-Hunek et al. 2020). Coatings produced using these agents exhibit significant variations in particle size, as shown in SEM microphotographs (Fig. 6).

Impregnation involves partially or fully filling the pores and capillaries in the near-surface layer of concrete, resulting in sealing as well as strengthening of the protected surface. A thin, discontinuous film may also form on the surface of the treated component. Impregnation typically employs materials based on plastic resins – epoxy, acrylic, polyurethane, polyester, as well as varnishes, mineral and/or polymeric substances. These resins often contain aliphatic hydrocarbons and volatile organic compounds (VOCs). Impregnation enhances the concrete's resistance to mechanical stresses, including abrasion, impact, thermal shock, freezing-thawing, even in salt-laden environments. It is considered a more durable solution, with protection lasting for several years.

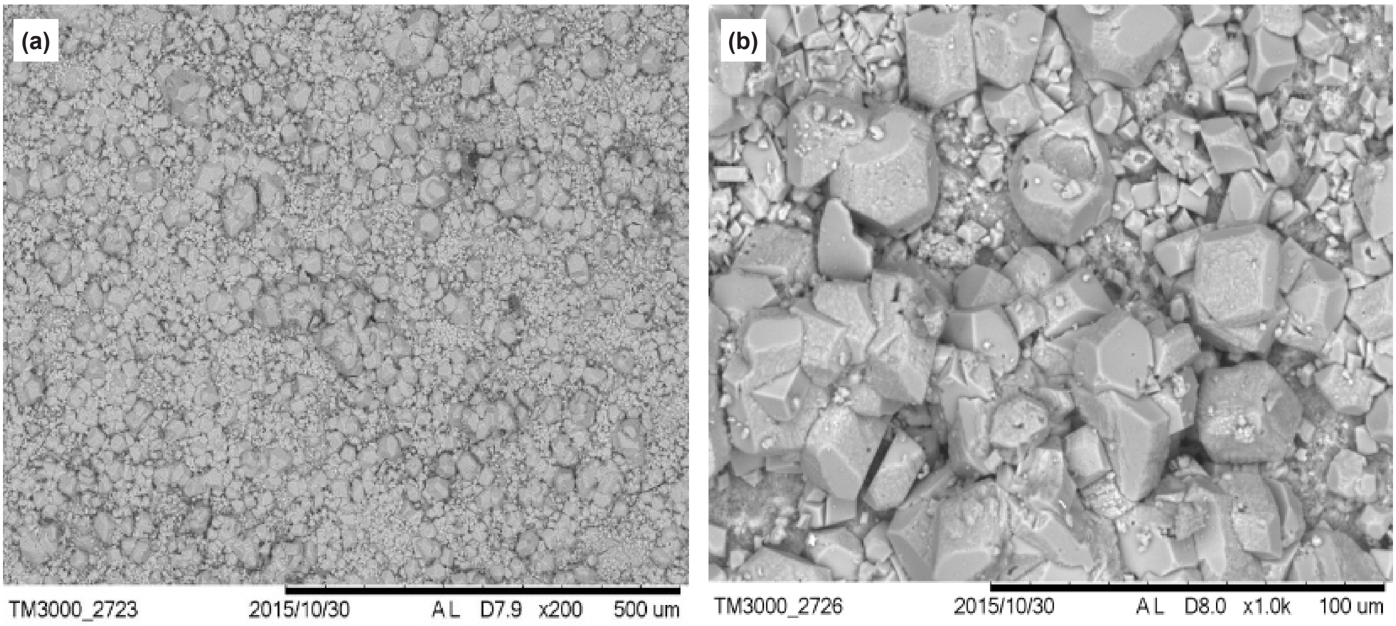


Fig. 6. Microtomography of silica gel formed from: a) silane, b) methyl silicone resin in aliphatic solvent (x1000).

A protective coating is formed using materials based on synthetic resins, such as epoxy, acrylic, polyester, polyurethane, epoxy-polyurethane, polycarbonate, polymer-cement bituminous, polymer-bitumen materials and others. These resins typically contain aliphatic hydrocarbons and volatile organic compounds (VOCs). The coating forms a continuous, airtight layer, usually no thicker than 5 mm thick, protecting the component from the effects of aggressive environments. It is also resistant to hydrostatic evaporation of liquids. Depending on the needs, the coating can be reinforced with fabric or mesh inserts, such as fiberglass. Chemical-resistant coatings can be glued or welded to plant plastic membranes, with the most common materials being high-density polyethylene (HDPE) films, polyvinyl chloride (PVC) films, and polyisobutylene films. These materials are used where complete protection against the penetration of substances is required, while also allowing for easy cleaning of concrete surfaces.

Effect of anti-corrosion coatings on concrete durability

Since water is the primary factor contributing to the degradation of concrete, high resistance to water ingress is a key aspect of anti-corrosion coatings. One crucial factor in water protection is the variation in water diffusion coefficients between the concrete and the coating. The water diffusion coefficient of concrete typically ranges from 10^{-9} to 10^{-12} $m^2 s^{-1}$ (Mihaljevic and Chidiac, 2022). In contrast, diffusion coefficients in many coatings are much lower, often ranging from 10^{-11} to 10^{-14} $m^2 s^{-1}$. When a coating with a lower water diffusion coefficient than the concrete is applied, the diffusion of water into the concrete is greatly reduced.

Oligomeric siloxanes, alkyl alkoxy-silanes, methyl methacrylate, silanes with an acrylic top layer, and epoxy coatings have shown significant success in reducing water absorption in concrete. In the case of hydrophobic impregnations, siloxanes and silanes have demonstrated a significant ability to reduce water ingress when the water pressure is less than 120 kgf/m² (Medeiros and Helene, 2009). This suggests that these agents are effective when the pressure of water acting on the surface is known. In most cases, surface treatments can be effectively prevent the penetration of chloride ions. A study by Almusallam et al. (2003) found that acrylic and polyurethane coatings were approximately 10-fold more effective at preventing chloride ion diffusion compared to non-coated concrete. Epoxy coatings proved to be twice as effective as chlorinated rubber coatings. Additionally, nanocomposite coatings typically outperform conventional polymer coatings in terms of performance.

Siloxane and silane effectively regulate the moisture level at the concrete surface without reducing air permeability or the diffusivity of CO_2 in the material. While most surface treatments can reduce concrete carbonation, silane and siloxane treatments do not significantly affect carbonation (Aguiar and Júnior, 2013). The thickness of the coating plays a very important role, and both interface properties and pore distribution can affect sulfate penetration. Protective coatings have been found to slow moisture penetration during freezing-thawing cycles. Basheer and Cleland (2006) reported that silane application could double the number of freezing-thawing cycles before concrete started to crack by two-fold. Other researchers have confirmed that epoxy resin and silane are effective formulations for protecting concrete against sodium sulfate, although their mechanisms differ. Epoxy resin forms a thick coating on the surface of concrete that prevents sulfate penetration, while silane penetrates the concrete and chemically reacts with the pores to create a hydrophobic surface (Suleiman et al. 2014). In contrast, aqueous acrylic resin is not resistant to sulfate attack.

Numerous studies have shown that hydrophobic protective coating using silane significantly reduces de-icing salt flaking (Thissen et al. 2024). The silane coating prevents the cryogenic suction phenomenon responsible for salt flaking. Studies have also shown that temperature and UV radiation significantly affect the performance of protective coatings. Levi et al. (2002)

found that fluorinated polymers, silicone, and silane reduced concrete water absorption by 50%, 90% and 50%, respectively, after UV aging. Notably, the silane-based waterproof coating lasted twice as long as the cement-based coatings and nearly twenty times longer than epoxy and polyurethane coatings. Performance differences were observed across manufacturers, even for coatings of the same type. Therefore, it is recommended not to select a coating for moisture protection based solely on one criterion, rather, individual tests should be conducted before application (Thissen et al. 2024).

Inorganic or organic nanocomposite coatings have recently attracted significant attention due to their effectiveness in increasing concrete durability. For instance, the incorporation of SiO_2 nanoparticles improves the material's microstructure, achieving a high contact angle (CA) of up to 156° (She et al. 2020). However, prolonged exposure to water can degrade the chemical bonds between the concrete surface and organosilane molecules, causing the loss of hydrophobic groups. To improve the properties of nanoparticle coatings, researchers have introduced polydimethylsiloxane (PDMS), a biocompatible and non-toxic material. Facio and Mosquera (2013) developed a superhydrophobic coating by condensing silica oligomers with PDMS, which proved effective in protecting concrete surfaces. Other studies have applied superhydrophobic coatings to concrete using silicone and SiO_2 nanoparticles (Thissen et al. 2024). These coatings showed excellent chemical stability and resistance to ultraviolet (UV) radiation. However, excessive amounts of polymer or nanoparticles can reduce hydrophobicity or destroy the polymer. Furthermore, researchers have demonstrated that the inclusion of nanomaterials, such as TiO_2, $CaCO_3$, and SiO_2, into silicone emulsions improved resistance to chloride ions by 76%. The addition of nanocarbon to epoxy coatings contributed to 66% increase in chloride diffusion resistance (Thissen et al. 2024).

Superhydrophobic coatings for concrete are not limited to nanosilica. Coatings made from graphene oxide and silane have been developed, with a contact angle (CA) of up to 165.5° on concrete surfaces (Thissen et al. 2024). While other nanoparticles such as iron tetroxide, zinc stearate, carbon nanotubes, and silver nanoparticles have proven effective in creating superhydrophobic coatings, there has been limited detailed research on superhydrophobic concrete. This may be due to the high cost of these additives and challenges associated with their practical application on large-scale concrete structures (Wu et al. 2022). An alternative approach involves polymer-modified cementitious coatings (PCCs), which are two-component, water-based coatings composed of cement and polymer emulsion. PCCs provide an environmentally friendly option as they are free from toxic VOCs, because water acts as the solvent. Additionally, these coatings can be applied to wet or medium-wet substrates, a feature that is not suitable for coatings containing VOCs, such as epoxy resins and silanes (Wu et al. 2022). PCCs are applied using a brush and offer the benefit of combining the flexibility of polymers with the rigidity of cement, which enhances the concrete's strength and permeability while filling cracks caused by higher elastic modulus. Some of these superhydrophobic coatings also exhibit self-repair capabilities.

Microbial corrosion is a significant type of corrosion that greatly affects the longevity and safety of concrete structures. Microorganisms are capable of producing a range of harmful substances, including volatile organic compounds, sulfides, and acids, making protection against them as crucial as protection against water. Recent studies have shown that a 2D coating made of hexagonal boron nitride and zinc oxide can effectively combat biological corrosion in concrete buildings, thereby enhancing their durability under unfavorable conditions (Zhang et al. 2024). The coatings have also shown antibacterial properties.

The choice of solvent in a formulation is important. Hydrocarbon solvent-based formulations are generally more efficient (Barnat-Hunek et al. 2020). Medeiros and Helene (2009) proved that siloxane and silane in an organic solvent were more effective at reducing water penetration compared to aqueous solvents. This enhanced performance was attributed to deeper penetration of the formulation into the concrete, resulting from the reduced viscosity of the solvent.

The use of anti-corrosion coatings is not limited to making concrete hydrophobic. Superhydrophobic coatings can enhance a range of surface properties, with various materials being utilized for specific improvements (Thissen et al. 2024):

- self-breathing: silane, PDMS, nanosilica,
- anti-icing: silane, nanosilica,
- self-cleaning: silane, TiO_2 nanoparticles,
- UV-protective: TiO_2 nanoparticles, SiO_2, perfluorodecyltriethoxysilane,
- slip resistance: polytetrafluoroethylene, diatomaceous earth, polytetrafluoroethylene,
- oil resistance: PDMS, ethoxysilane oligomers,
- protection against biological corrosion: calcium carbonate on magnesium-neodymium alloy (Mg-Nd),
- wear resistance: silane, TiO_2 nanoparticles, SiO_2,
- antibacterial properties; hexagonal boron nitride and zinc oxide, copper-modified calcium silicate hydrates.

Recent experimental studies have evaluated the effectiveness of anti-corrosion coatings applied to concrete surfaces. The promising results from these studies have contributed to the development of various commercially available concrete surface treatment methods. However, a wide range of options presents a challenge in selecting the most suitable treatment method. A review of the literature showed the need to eliminate toxic solvents containing VOCs. Considering this, the authors propose incorporating higher fatty acids, such as stearic acid (STA) and methyl esters (ME), in future studies.

Stearic acid

Stearic acid (STA) is an organic chemical compound and one of the saturated fatty acids. It is commonly referred to as octadecanoic acid. Molecular formula – $C_{18}H_{36}O_2$ (Fig. 7), alternate formulas: $C_{17}H_{35}COOH$, $CH_3(CH_2)_{16}COOH$, $CH_3–(CH_2)_{16}–COOH$, molar mass: 284.48 g/mol, boiling point 361°C, density 941 kg/m³, insoluble in water, melting point 67 - 72°C (Hassa et al. 2004).

Stearic acid (STA) is an 18-carbon saturated fatty acid found in many fats and oils of animal and vegetable origin (Fig. 7). Naturally, stearic acid occurs as a mixture of triglycerides or fat combined with other long-chain acids and as a fatty alcohol ester. It is obtained by hydrolyzing animal

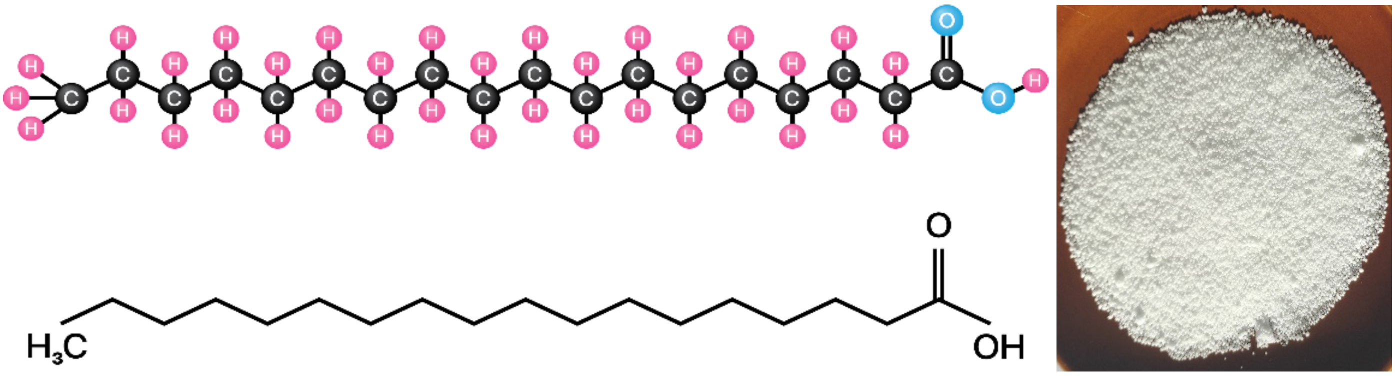


Fig. 7. Stearic acid $C_{18}H_{36}O_2$ (own elaboration)

fats at high temperatures and under increased pressure. $C_{18}H_{36}O_2$ can be derived through hydrolysis from animal fats under these conditions. Stearic acid is the second most abundant fatty acid (after PA) in foods, especially in coconut oil, butter and meat. Due to its versatility and low cost, stearic acid is widely used across various industrial sectors, making it one of the most important fatty acids used in industry. Specifically, stearic acid is used for protective coatings due to its amphiphilic nature and low cost. Stearic acid (STA, $CH_3(CH_2)_{16}COOH$) is an environmentally friendly material that enables superhydrophobicity. Its molecules form self-assembled monolayers (SAMs) due to their amphiphilic nature, facilitating the production of hydrophobic coatings on hydrophilic surfaces. Nevertheless, stearic acid remains solid at room temperature and is insoluble in water posing challenges for achieving uniform dispersion in concrete under these conditions.

Alternative methods of adding stearic acid to concrete have been explored. For example, stearic acid derivatives (including zinc stearate and calcium stearate), as well as stearic acid-coated powders are employed. However, powders derived from stearic acid exhibit high hydrophobicity or even superhydrophobicity, making it challenging to achieve uniform dispersion in water. Lei et al. (2020) created superhydrophobic concrete with excellent corrosion resistance and consistent mechanical strength by incorporating STA during molding. Similarly, Liu et al. (2020) developed durable superhydrophobic concrete using an STA emulsion. Their findings indicated that the resulting coating demonstrated effective superhydrophobic and self-cleaning characteristics, with a water contact angle greater than 158°. In the work of Feng et al. (2019), the authors used a 30% stearic acid emulsion in aqueous form as a hydrophobic additive to cement mortar to enhance impermeability. The modified mortars exhibited internal hydrophobicity. Both the surface and structure of the mortars were hydrophobic and a CA of water was greater than 130°. These hydrophobic mortars were mechanically durable and retained their hydrophobicityeven after abrasion, cutting and scratching. Additionally, the mortars demonstrated improved chloride ion (Cl^-) corrosion resistance and greatly reduced water absorption in comparison to the control sample.

The mortars exhibited a decrease in flexural and compressive strengths by 20% and 16%, respectively, compared to the control sample. Despite this reduction, their value remained high at 8.12 MPa and 36 MPa, respectively.

In a different research project, metakaolin modified with stearic acid was utilized to enhance the sulfate resistance of cement-based materials, mitigating the negative impacts of the conventional hydrophobic modifiers on the mechanical characteristics of the cement mortar. The acid-modified metakaolin reduced the transport of sulfate ions in the mortar. During the initial phases of corrosion, the higher compressive strength resulted from the continued hydration of the cement. However, a subsequent decrease in strength occurred due to sulfate erosion. The relative dynamic elastic modulus of the specimens initially increased and then fell as corrosion time progressed. This behavior was primarily attributed to the reduced water absorption of the cement, caused by the hydrophobic nature of the stearic acid-treated metakaolin.

An interesting study was conducted by Yang L. et al. (2024), who developed a new hydrophobic modifier to enhance the longevity of a cement-based materials while simultaneously avoiding a significant reduction in compressive strength. The hydrophobic admixture consisted of stearic acid (0%, 1%, 3%, 5% by weight of cement) and mica powder. Compared to non-modified mortar, the samples with 3% and 5% admixture exhibited water absorption reductions of 56% and 64%, respectively. With 5% STA content, the water contact angle of the mortar reached 102°. After 60 freeze-thaw cycles in a salt solution, the reference sample showed significant surface peeled, whereas the samples containing STA exhibited only localized surface spalling, with the smallest amount of spalling observed in samples with the highest STA content. However, with 5% STA content, the mortar experienced an increase in porosity and a decrease in strength, leading to more pronounced flaking of the sample. Inadequate bonding between the hydrophobic STA and the mortar may have been the primary cause of local damage in the sample, contributing to the decrease in compressive strength of the mortar. Using a small amount of STA did not significantly impact the compressive strength due to sufficient bonding force between the mortar matrix and the STA.

Moreover, an adequate quantity of stearic acid appeared to enhance cement hydration and boost the mortar structure density. At a 3% STA content, the benefits and drawbacks balanced each other, resulting in a negligible change in the sample's compressive strength. In contrast, the higher amount of stearic acid resulted in a greater release of free water and the formation of larger pores, causing an increase in mortar's porosity and a decrease in strength. SEM microphotographs showed that the reference sample had numerous wide cracks, large holes, and abundant ettringite needles within the cracks. Conversely, the sample with 3% STA showed fewer ettringite needles and smaller cracks, with a compact C-S-H structure. This further confirming that STA can reduce ettringite formation as well as slow crack formation and development.

Mica and STA were also used to develop a new hydrophobizing additive. The results revealed a significant 36% reduction in water absorption, which substantially decreased sulfate concentration. The authors demonstrated that the STA additive acts as a hydrophobic coating, exerting a bidirectional water transport-inhibiting effect. It was found that the STA-modified mortar becomes hydrophobic, as the hydrophobic groups of STA reduce the free surface energy of the mortar pore structure. In terms of mechanical performance, the STA-modified mortar had better mechanical properties than the reference mortar. The reference sample showed extensive salt-induced cracking and peeling, whereas no cracking was observed in the sample with 5% STA, even after 180 days of testing. The samples with 5% STA showed a 14.2% increase in compressive strength after the salt test. The authors proved that the STA additive acts as a hydrophobic coating.

Cunha et al. (2022) investigated the properties of cement mortars containing ceramics and paraffin waxes. They found that both flexural and compressive strength decreased compared to the control mortar, with reductions becoming more pronounced as paraffin content increased. This decline was attributed to poor adhesion between paraffin and the mortar matrix. Paraffin-polyglycerol fatty ester, a non-ionic surfactant composed of polyglycerol and fatty ester, exhibits excellent hydrophilicity due to the presence of hydroxyl groups (Salar-Behzadi et al. 2020). The hydroxyl groups are beneficial for enhancing interfacial bonding efficiency and improving water retention due to their hydrophilic nature. However, studies by Wang et al. (2024) have shown that paraffin-polyglycerol fatty ester delays hydration and causes dormancy due to its hydrophobicity. Other studies have shown that the addition of organic coating materials to cement can reduce mechanical properties of cementitious materials (Cunha et al. 2020). These effects may result from the formation of complexes, such as calcium oleo-aluminate complexes, which inhibit the development of calcium silicate hydrate (C-S-H) gel (Albayrak et al. 2005). Oils comprising saturated aliphatic hydrocarbons or saturated fatty acids (e.g. myristic acid, lauric acid, and stearic acid) have a minimal effect on hydration but still affect strength through changes in the microstructure (Albayrak et al. 2005).

Hydrophobic coatings have received much attention in metal surface engineering. Recently, Li et al. (2024) developed a coating combining Fe_3O_4 and TiO_2 nanoparticles modified with hydrophobic stearic acid (SA) for oil-water and emulsions separation. The coating exhibited water and oil CAs of 159.8° and 154.4°, respectively, demonstrating superhydrophobicity. It also showed excellent self-cleaning properties and good mechanical strength. In a study by Loperen et al. (2024), stearic acid-based anti-corrosion composite coatings were created to improve the corrosion resistance of AZ91D Mg alloy. The composite coating demonstrated strong adhesion and a dense structure, with surface hydrophobicity influenced by STA concentration. The effect of an aggressive environment on the coating morphology was studied, revealing no significant surface changes after 24 hours of immersion in Ringer's solution. The coating retained its hydrophobic properties, maintaining CA above 90°, even after 24 hours of immersion, and effectively blocking contact between the substrate and the corrosion solution. Liu et al. (2020) successfully developed a superhydrophobic and corrosion-resistant coating on AZ31B Mg alloy by electro-depositing a CeO_2 film and then immersing it in stearic acid.

Other researchers have developed new coatings for steel using a nickel-based Ni625 alloy and stearic acid. The addition of stearic acid significantly increased the presence of long-chain carboxylate molecules, enhancing the hydrophobicity of the coating. As a result, the water CA on the coated surface increased from 137° to 151° (Zhang et al. 2023).

Hydrophobic coatings made from STA and soybean oil are increasingly used in the textile industry. In a study by Xu et al. (2022), cotton fabric was coated with a thermosetting material derived from epoxidized soybean oil and then modified with stearic acid (STA). The modified fabric showed superhydrophobic properties, with a CA of 159.7°. Even after 1,000 abrasion cycles or 30 wash cycles, the coating a CA above 154°, showing excellent mechanical durability.

The cited tests confirm the hydrophobic nature of coatings formed with STA on various materials. STA can prevent water penetration, even in the case of cracks, thereby enhancing the durability of materials in corrosive environments. For hydrophobic concrete, the hydrophobic coatings inhibit the entry of corrosive ions, even when there are a significant number of pores.

Methyl esters

Fatty acids are carboxylic acids with long hydrocarbon (aliphatic) chains that can be either saturated or unsaturated (containing double bonds). Each fatty acid molecule contains a carboxyl group (-COOH) located at one end of the chain (Fig. 8).

Under specific conditions, the carboxyl group (-COOH) can lose a proton, creating a negatively charged functional group capable of interacting with positively charged ions or functional groups (Yuan et al. 2014). Due to the high phase transformation temperature, corrosiveness, unpleasant odor, and sublimation occurring during the heating of fatty acids, some researchers have opted to use their derivatives, such as fatty acid esters. These esters are produced by esterifying fatty acids with alcohols.

The general esterification reaction is shown as equation (1) (Yuan et al. 2014).:

$$R-COOH+R'-CH_2OH \; ^{(H_2SO_4)} \quad R-COOH_2-R'+H_2O \qquad (1)$$

Methyl ester residue (MER) is a byproduct generated in manufacturing plants. During the vacuum distillation of crude palm or rapeseed oil, glycerin tar and MER accumulate at the base of the distillation column. Although MER contains valuable components like residual oil and ester, the high costs of extraction and recycling make it less economically viable. Consequently, some industrial waste ends up in landfills or is dumped into oceans, raising significant environmental concern. Hence, researching affordable ways to recycle and reuse MER is necessary. Currently, MER has not been explored for use in building materials. Fatty acids are appealing for such

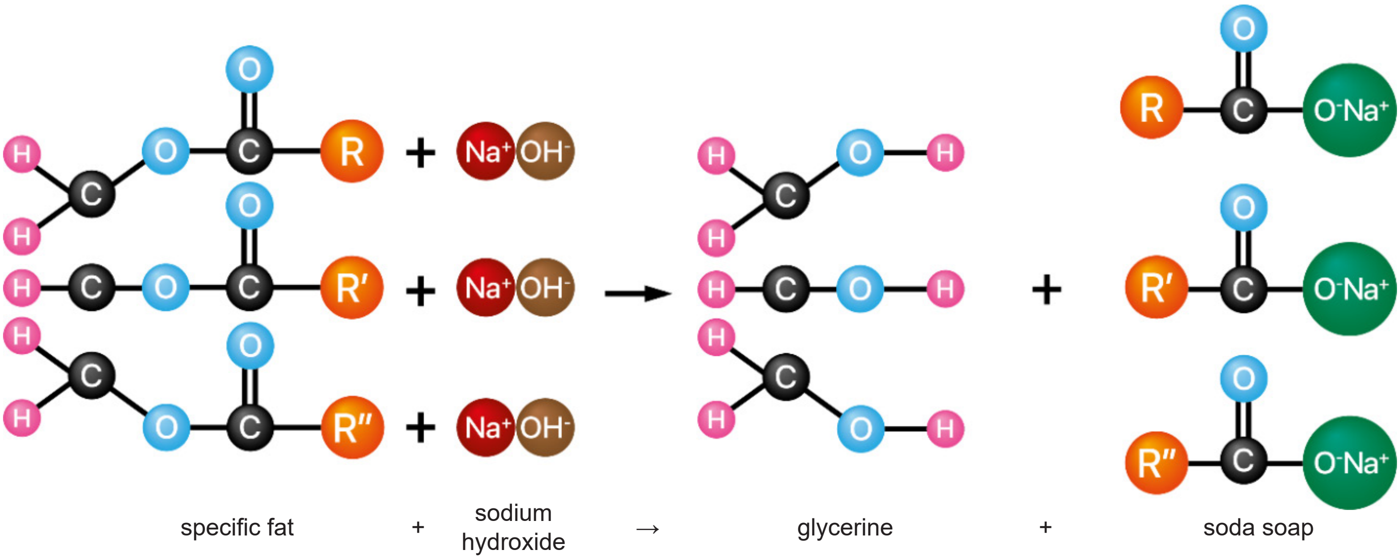


Fig. 8. Fatty acid methyl esters (own elaboration).

application because they are derived from fossil fuels, are less combustible than paraffins, and, in some cases, do not need microencapsulation (Yuan et al. 2014).

The findings of Zajezierska (2016) indicate that using a base oil composed of rapeseed oil (70%) and synthetic ester oil (30%) in lubricant synthesis enables the production of biodegradable products. These products exhibit good resistance to aging processes, comparable to conventional bearing greases made from petroleum oils with predominantly paraffinic structures. The chemical characteristics of fatty acids make them appropriate for incorporation into concrete mixes. Their carboxyl groups can bond with positively charged colloidal particles in concrete, similar to the mechanism of polycarboxyl ether (PCE), a widely used superplasticizer (Cellat et al. 2015).

Hydrophobic substances such as animal fats, vegetable oils, emulsions, fatty acid soaps, siloxanes, and silanes, alter surface tension or surface energy within cracks and pores. This modification increases the contact angle of liquids, thereby reducing water infiltration. At present, there are no standardized methods for measuring fatty acids in cement and concrete. Fatty acids can differ based on their placement within the glyceride molecule, chain length, and the number and location of double bonds. The variations result in diverse impacts on cement slurry behavior. With growing awareness of sustainability, researchers have explored the use of organic waste materials as alternative binders in building materials. For example, spent motor oil, spent cooking oil, glycerin tar, and MERs have been used in tile production (Yaras et al. 2019). Tiles made with these binders exhibited resistance to water, sulfuric acid, and sodium chloride solutions. During fire resistance testing, a portion of the sample subjected to fire sources ignited, but the flames did not spread to other areas of the specimen. Despite being derived from oil-based components, most of them polymerized when exposed to heat during the treatment process. As a result, MER was found to be neither ignitable nor flammable. The material self- extinguished when removed from fire source and was classified as an incombustible substance according to EN 13501-01 standards.

Barnat-Hunek et al. (2023) evaluated the effectiveness of new concrete surface hydrophobizing agents. These agents were composed of organic oils, siloxanes, water-soluble silanes, and/or tap water. The formulations aim to reduce the environmental impact of volatile organic compounds (VOCs), as they primarily consist of biodegradable components. Research has demonstrated that elevated levels of fatty acids derived from vegetable oils and the glycerin phase are suitable for use in concrete hydrophobizing agents, satisfying the specified performance criteria. Vegetable oil-based industrial substances showed a minimum of 95% natural biodegradability. Hydrophobization with methyl esters reduced concrete absorbability by up to 52%. Silane and higher fatty acids formed a tight surface layer that prevented external water absorption but also blocked water vapor escape, potentially causing internal deterioration. Watertightness tests revealed that methyl ester-based agents were highly effective, achieving a water penetration depth of only 6 mm under pressures of up to 0.8 MPa (classified as W8 watertightness per PN-B-06250. The standard concrete experienced a reduction in weight of 1.8%, following frost resistance test. Frost resistance test indicated significant improvements with hydrophobization, where weight loss was reduced by 33 to 72% compared to reference samples. While salt crystallization occurred in concrete treated with glycerin-based agents, no structural damage was observed. By contrast, untreated control samples showed a weight reduction of 0.8%. Wang et al. (2015) showed that water droplets on fatty acid-coated surfaces rolled off, removing dirt and leaving clean trails. All samples exhibited superhydrophobic properties with excellent self-cleaning behavior. The carbon chain of organic acids affected the wettability and corrosion resistance results. Fatty acid-treated samples achieved the highest contact angle (164°) and exceptional corrosion resistance (99.98%) in NaCl solution.

Recently, epoxy coatings reinforced with microcapsules containing tung oil have been introduced for treating steel in concrete. Tung oil absorbs oxygen from the air and rapidly forms a solid, impermeable, and waterproof coating, effectively protecting steel from corrosion. The addition of microcapsules to the epoxy coatings results in a rougher surface on rebar compared to uncoated rebar. Coated rebar exhibits greater pullout strength than uncoated rebar. In addition, corroded bars with the coating show 68% higher tensile strength compared to corroded control bars. For example, epoxy resins derived from soybean oil (Hu et al. 2023), or clove oil (eugenol) (Tian et al. 2024) show mechanical, thermal and fire resistance properties comparable to petroleum-based bisphenol A epoxy resins.

Research by Johansson K. and Johansson M. (2007) indicated that the amount of traditional solvents in paints can be reduced by replacing them with methyl esters derived from rapeseed, without compromising the mechanical properties of the paint film. Wang et al. (2019) found that cardanol methacrylate and triethoxysilane functionalized cardanol can serve as reactive soybean oil-based thinners for alkyd coatings. Other renewable sources, such as palm oil, grape seed oil, corn oil, canola oil, peanut oil, and olive oil, have also been used to prepare ultraviolet-curable coatings (Su et al. 2020). Zweep (2014) used vegetable oil-based polyols, lactic acid esters, and vegetable oil-based plasticizers to produce water-based polyurethane paints, eliminating VOCs entirely (Zweep, 2014).

Conclusions

As a result of environmental concerns, researchers are seeking eco-friendly alternatives to organic coatings with VOCs, aiming for sustainability. Preserving concrete with surface coatings is a complex challenge that involves a combination of chemical and physical mechanisms. Key findings from the literature review are summarized below. The impact of hydrophobic impregnations and organic coatings using higher fatty acids on the mechanical characteristics of concrete has not been extensively researched. In most cases, protective coatings are effective in preventing the penetration of aggressive substances. Silicate and higher fatty acid-based coatings offer improved protection compared to siloxane and silane, which have limited capability to prevent CO_2 penetration. In general, organic coatings offer superior resistance compared to hydrophobic silane- and waterglass-based coatings. However, acrylic coating fail to protect against physical attack by sulfates due to their brittle nature. Most studies focus primarily on protecting concrete from damage caused by freeze-thaw cycles, but the results remain inconclusive.

Epoxy coatings and waterproof silane coatings have been proven effective in resisting salt flaking. However, there are no studies on the use of coatings with the addition of higher fatty acids for this purpose. Inorganic coatings, such as nanocomposites and silicate-based coatings, present an alternative to organic coatings containing VOCs. Nonetheless, nanocomposites, comprising materials like graphene, nanosilica, titanium dioxide, silver nanoxide, are expensive and unlikely to be widely used. Additionally, they are less flexible than organic coatings, which can cause coatings to crack.

The proposed method of modifying anti-corrosion coatings with biodegradable esters of higher fatty acids align well with the principles of sustainable development, particularly by utilizing agricultural waste materials in cooling towers. The development of coatings with higher fatty acids is an effective strategy to reduce VOC emissions in the petroleum-based resin and impregnation industry.

The review of the literature shows that it is necessary to further research into anti-corrosion coatings for concrete structures that incorporate biodegradable admixtures, such as higher fatty acids. These coatings show promising results in terms effectiveness, VOC reduction, environment impact, and cost savings. The authors' forthcoming research will explore these aspects in greater detail. Improved performance of these new products will depend on the optimized structure and properties of their surface layers, especially their durability.

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Redukcja lotnych związków organicznych (LZO) w żywicach epoksydowych poprzez stosowanie wyższych kwasów tłuszczowych z przemysłu rolniczego – przegląd

Streszczeanie: Wadą wielu powszechnie stosowanych impregnatów i żywic jest ich wysoka toksyczność, związana z obecnością szkodliwych węglowodorów aromatycznych i lotnych związków organicznych (LZO) w ich składzie. LZO stanowią stosunkowo dużą, ok. 30% część w przemyśle żywic syntetycznych. Jednym z pomysłów na redukcję lub wyeliminowanie LZO z produkcji żywic i farb jest stosowanie wysokiej jakości półproduktów i biodegradowalnych surowców. Przedstawiono perspektywę nowych podejść do ochrony powierzchni betonowych, obejmującą koncepcję wykorzystania w tym celu dwóch rodzajów wyższych kwasów tłuszczowych: kwasu stearynowego (STA) i estrów metylowych (ME). Ostatnie postępy technologiczne skupiają się na surowcach z olejów roślinnych do zastosowań przemysłowych. Wynika to z ich przydatności do przemysłowej produkcji środków, ponieważ zastępują one nieodnawialne węglowodory. Przytoczone badania potwierdzają hydrofobowy charakter powłok tworzonych przy użyciu STA i ME na różnych materiałach. Z analizy literatury wynika, że należy rozwijać badania nad powłokami antykorozyjnymi z domieszkami biodegradowalnymi, tj. wyższymi kwasami tłuszczowymi, ze względu na ich obiecujące wyniki w zakresie efektywności, redukcji substancji toksycznych (LZO) i ich wpływu na środowisko.