Fluorosilicone Surfactants: Properties, Synthesis Methods, and Industrial Applications

Qianyi Wang [1], Yilin Yang [2], Xinyue Zhang [3, *]

[1] NOIC Academy, Markham, Ontario, L3S 2H4, Canada

[2] TICC CUNZHI Academy, Shanghai, 201906, China

[3] Department of light Chemcial engineering, Nanjing Tech University, Nanjing, China

* Corresponding Author Email: xubin12333321@tzc.edu.cn

Abstract. Fluorosilicone surfactants have garnered significant attention in recent years due to their superior properties resulting from the integration of silicone and organic fluorine materials. Their unique molecular structure imparts exceptional attributes, including heat resistance, weather resistance, corrosion resistance, and hydrophobic and oleophobic characteristics. These properties render fluorosilicone surfactants indispensable in various industrial applications, such as the rubber industry, coatings, metal lubrication, and electronics. The chemical structure of fluorosilicone surfactants predominantly comprises Si—O—Rf and Si—C bonds. The Si—O—Rf bonds confer notable flexibility and reactivity, while the Si—C bonds provide enhanced stability. Among the synthesis methods for fluorosilicone surfactants, hydrosilylation and free radical polymerization are the most prevalent. Researchers are also investigating more environmentally friendly synthesis techniques to advance green chemistry. This article provides a comprehensive review of fluorosilicone surfactants, including their structural characteristics, physicochemical properties, synthesis methods, and applications. It emphasizes their extensive potential across various sectors, such as rubber, coatings, metal lubrication, and emerging fields. Furthermore, it explores future application prospects and the potential for environmentally sustainable development in novel industries.

Keywords: Fluorosilicone surfactants, silicone, organic fluorine materials, hydrosilylation, free radical polymerization.

1. Introduction

Surfactants are a group of substances that can significantly reduce the surface tension of a solution and modify interfacial properties when added in slight quantities [1]. One type of surfactant is the organosilicon surfactant, which contains a polysiloxane structure with a main chain consisting of Si–O–Si bonds. Given that the Si–O bond has much higher bond energy than both C–O and C–C bonds, organosilicon surfactants exhibit excellent heat resistance and good weather stability. In contrast, fluorosurfactants, which contain fluorocarbon chains (Rf hydrophobic groups), are formed by substituting hydrogen atoms in hydrocarbon chains with fluorine atoms [2]. This type of surfactant possesses exceptional chemical stability, weather resistance, oxidation resistance, and corrosion resistance, attributed to the very high C–F bond energy and short chemical bonds [2]. Additionally, fluorosurfactants exhibit unique "three high" and "two hate" properties, namely high surface activity, high thermal stability, high chemical stability, as well as hydrophobic and oleophobic characteristics [2].

Fluorosilicone surfactants can be synthesized by reacting organofluorine compounds with high surface activity with organosilicon polymers. This reaction typically occurs when fluorinated alkyl groups are located at the side chains or the terminal ends of polysilafluoranes [3]. This reaction mechanism allows fluorosilicone surfactants to integrate the advantages of silicon and organofluorines: the heat and cold resistance of silicones, and the weathering, oil, solvent, and corrosion resistance, alongside the lower surface energy of fluorines. This paper will discuss in detail the structural features, physicochemical properties, synthesis methods, application areas, and future developmental trends of fluorosilicone surfactants.

2. Overview of the Structural Features and Properties of Fluorosilicone Surfactants

2.1. Molecular Structure

The molecular structure of fluorosilicone surfactants can be classified into two primary categories. The first type involves a structure where silicon and fluorinated alkanes are connected by oxygen (i.e., the Si–O–Rf structure), typically prepared through the condensation of polysiloxanes containing silicon functional groups with fluorinated alkyl alcohols [2]. The Si–O chain possesses a flexible and curly configuration, enabling the introduction of silicon groups into the organofluorine structure to form interpenetrating network structures between silicon and fluorine polymers, thus enhancing the material's functionality [2]. Although these structures exhibit reduced stability in acidic and alkaline solutions, they are characterized by high reactivity and ease of synthesis [2]. The second type of structure involves the bonding of silicon and fluoroalkanes via Si–O bonds [2]. Fluorosilicone surfactants of this type demonstrate excellent stability under a wide range of pH and temperature conditions, making them the most prevalent category of fluorosilicone surfactants in contemporary industrial applications [3].

2.2. Key Physical and Chemical Properties

Organosilicon surfactants are capable of reducing the surface tension of water to approximately 21 mN/m, attributed to their remarkable surface activity and spreading properties. In contrast, fluorosilicone surfactants, owing to the highly reactive nature of fluorine atoms, can further reduce surface tension to 15–16 mN/m, even in low-concentration aqueous solutions, significantly outperforming non-fluorinated surfactants [2]. For instance, the fluorosilicone surfactant developed by Hideo Sawada et al. exhibits not only excellent solubility in water but also performs effectively in aromatic solvents such as benzene, toluene, and m-xylene [4]. This surfactant significantly reduces the surface tension of these solvents and lowers the surface tension of water to 20.6 mN/m [4]. These outstanding physical and chemical properties endow fluorosilicone surfactants with vast potential for applications across various fields.

3. Preparation Methods

The fluorosilicon surfactant, composed of silicon and fluorinated alkanes connected through Si—C bonds, represents the primary variety in the industrial production of fluorosilicon surfactants. Common preparation methods include hydrosilylation and free radical polymerization [5].

3.1. Hydrosilylation

Fluorinated alkenes or fluoroalkyl alkene ethers can undergo hydrosilylation with hydrogen-containing silanes or silicone oils to synthesize fluorosilicone surfactants. In industrial production, Speier and Karstedt catalysts are commonly used for these hydrosilylation reactions. Depending on the chosen raw materials, either ionic or non-ionic surfactants can be produced. The additional reaction between Si-H and C=C bonds typically occur in the presence of a catalyst. Hydrosilylation is a widely employed method for preparing fluorosilicone polymers [6]. This technique is not only simpler but also more efficient compared to other methods discussed later. However, the reliance on toxic metals as catalysts raises concerns about its environmental impact and restricts the diversity of fluorosilicone polymers that can be synthesized [7].

1) Fluorosilane is produced through the hydrosilylation reaction between fluorinated alkenes and hydrogenated silanes, followed by hydrolysis and condensation to form fluorosilicone surfactants (as shown in Figure 1).

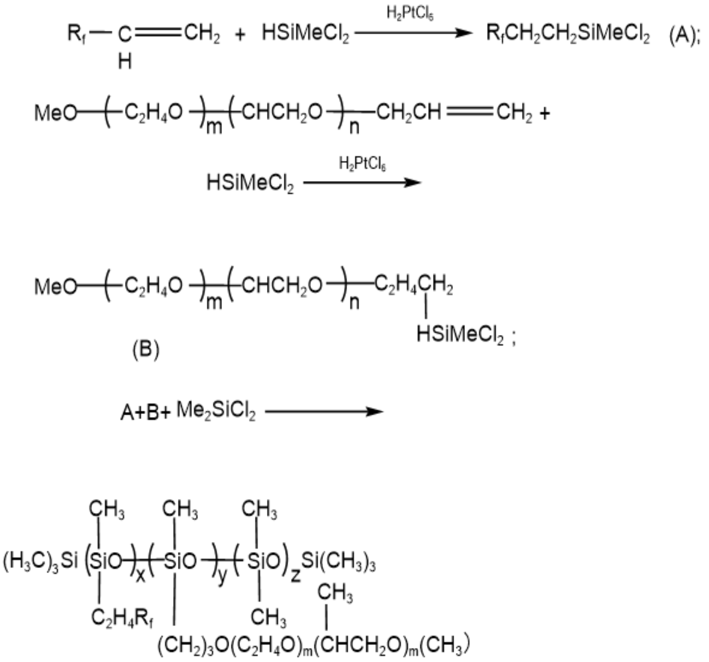


Fig. 1 Fluoroalkyl alkene ethers hydrosilylation reactions with hydrogen-containing silicone oils [5]

2) Fluoroalkyl alkene ethers undergo hydrosilylation reactions with hydrogen-containing silicone oils, or fluorinated olefins and terminal vinyl polyethers are hydrosilylation-reacted with hydrogen-containing silicone oils to produce fluorosilicone surfactants (Figure 2) [5].

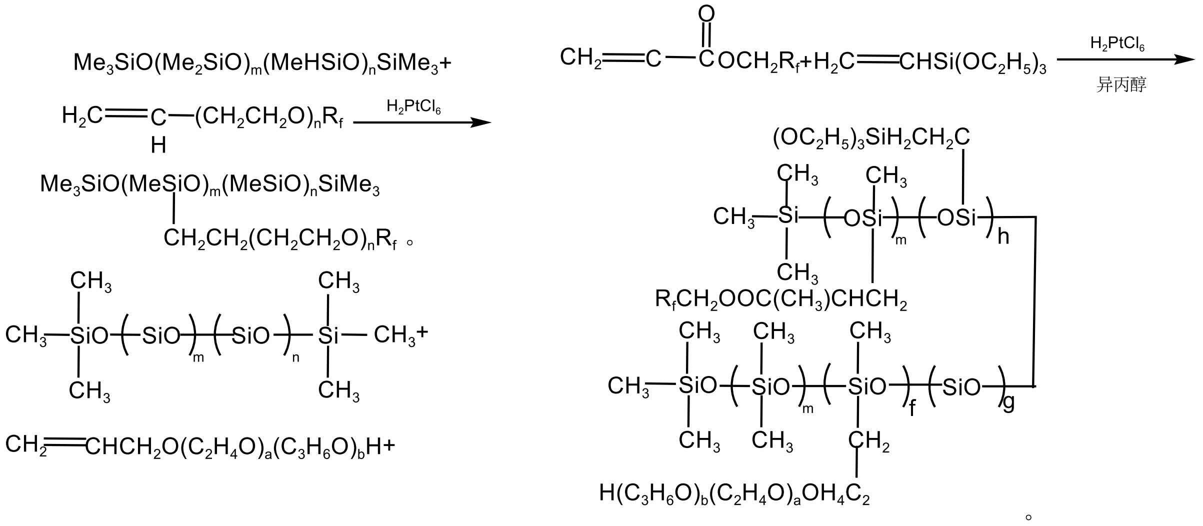
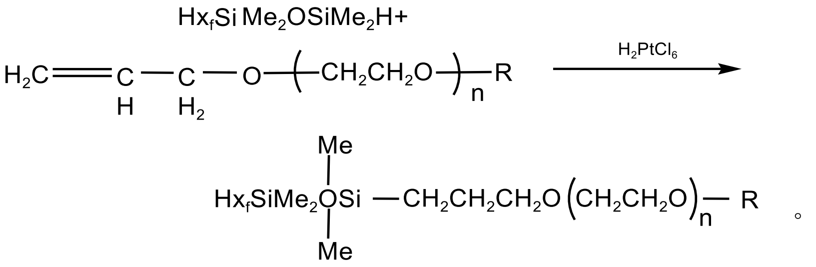


Fig. 2 Fluoroalkyl alkene ethers hydrosilylation reactions with hydrogen-containing silicone oils [5]

3) Fluorosilicon compounds with Si-H bonds and polyoxyethylene containing allyloxy groups undergo a silicon-chloride addition reaction to produce fluorosilicon nonionic surfactants (as shown in Figure 3) [8].



其中,R=H、CH_3、$COCH_3$、$Hx_f=CH_2CH_2CF$、$CH_2CH_2CF_2CF_2CF_2CF_3$

Fig. 3 Fluorosilicon compounds with Si-H bonds undergo a silicon-chloride addition reaction [5]

3.2. Free Radical Polymerization

Fluorosilicone surfactants can also be synthesized through the copolymerization of fluoroolefins or fluoroalkyl alkenyl ethers with free radicals containing double-bonded silanes. Based on the physical state of the reaction system, free radical polymerization can be categorized into emulsion polymerization, solution polymerization, and bulk polymerization. These radical polymerization processes are often accompanied by chain transfer reactions [5]. While controlling the structure of free radical reactions can be difficult, controlled radical polymerization can be achieved with the use of specific initiators.

Compared to hydrosilylation, free radical polymerization does not require the use of heavy metal catalysts, making it a more environmentally friendly option. Additionally, free radical polymerization ensures near-complete polymerization of the monomers and offers broader applicability. The process accommodates a wide range of monomers, operates under lower temperature conditions, and is relatively easy to control. However, polymers produced by this method tend to have a higher molar mass but with a broader molar mass distribution. Free radical polymerization techniques can be further divided into bulk polymerization, emulsion polymerization, solution polymerization, and even supercritical carbon dioxide polymerization. This paper focuses primarily on the first three methods.

1) Emulpolymerization

Firstly, a fluorinated monomer was synthesized using perfluorooctanoic acid (PFOA) as the fundamental raw material (as shown in Figure 4).

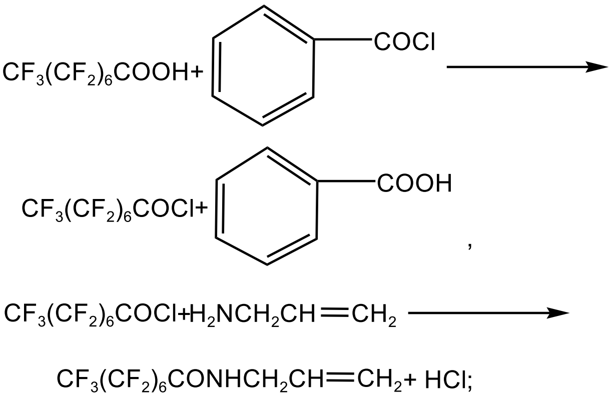


Fig. 4 Synethesis of fluorinated monomer using perfluorooctanoic acid [5]

Then the hexomethylcyclic tetrasililoxane and this fluorinated monomer were added to produce the cyclosiloxane containing perfluorooctyl (as shown in Figure 5) [5]

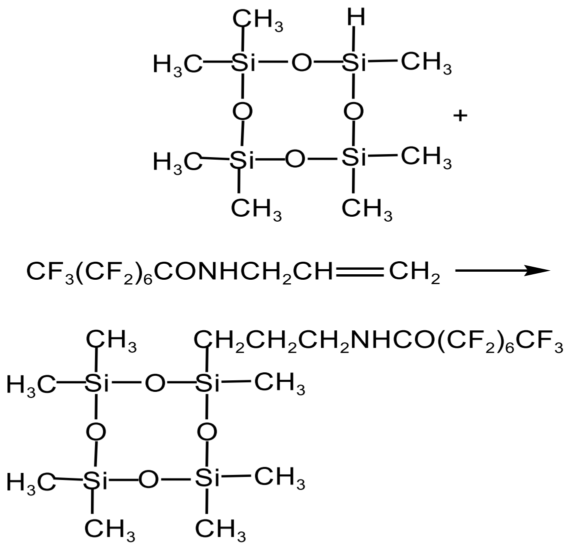


Fig. 5 Production of the cyclosiloxane containing perfluorooctyl [5]

2) Solution polymerization

Adding $CH_3(CH_2)_{11}SH$ as the chain transfer agent and methylethyl ketone as a solvent, copolymerization of fluoroalkyl acrylate and $CH_2{=}C(CH_3)CO_2(CH_2)_3Si(OSi(CH_3)_3)_3$ with low surface free energy and better hydrophobicity (as shown in Figure 6).

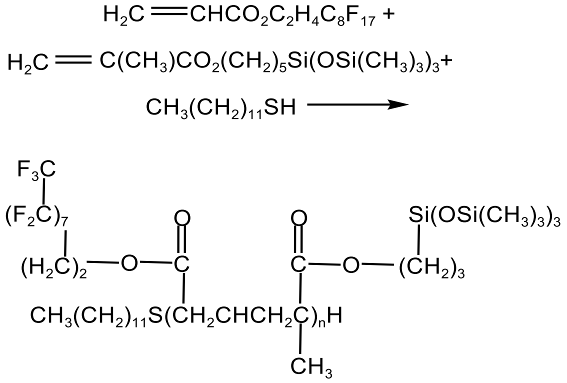


Fig. 6 Solution polymerization process [4]

3) Mass polymerization

1,3-double (2-hydroxyl hexafluoroisopropyl) benzene, dichlorodimethylsilane($Cl_2Si(CH_3)_2$), (methyl) acryryl chloride, and a homopolymer with azobisisobutyronitrile (AIBN) as initiator; or mass polymerization with methyl acrylate (MA)(as shown in Figure 7) [5].

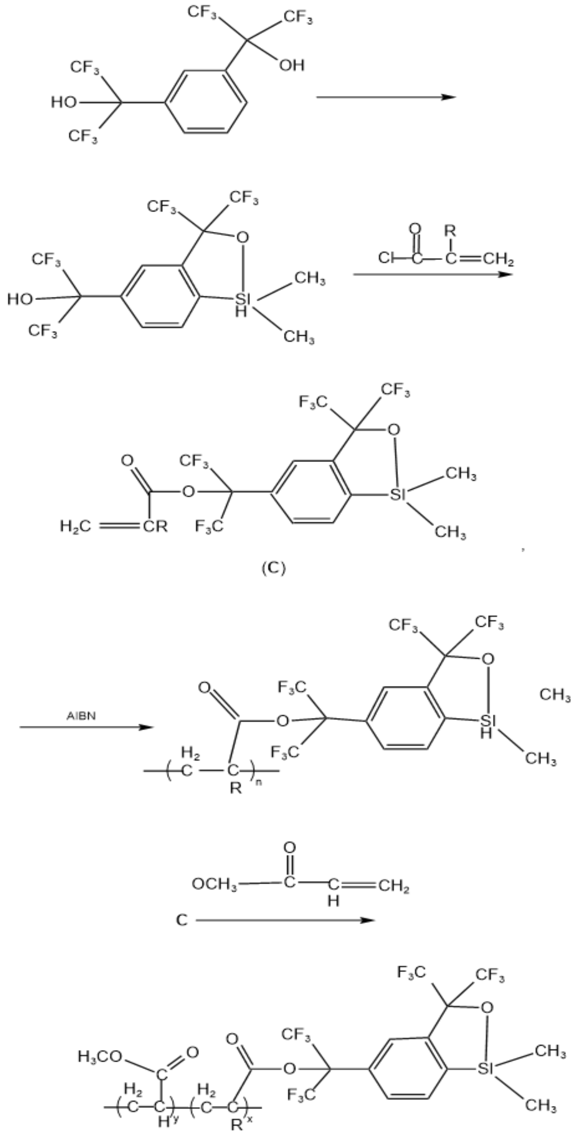


Fig. 7 Mass polymerization process [5]

3.3. Other Synthetic Methods

3.3.1. Organometallic compound

1) Grigner's reagent method

Fluoroalkyl halothane or haloaromatic hydrocarbons react with magnesium to form a Grignard reagent, which subsequently reacts with alkylchlorosilane in an oxygen-free environment, typically under nitrogen (N_2) protection [7]. This process yields fluorosilane, which can then undergo hydrolysis and polycondensation reactions to synthesize fluorosilicone surfactants (as illustrated in Figure 8).

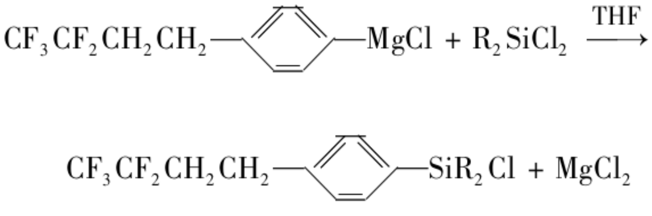
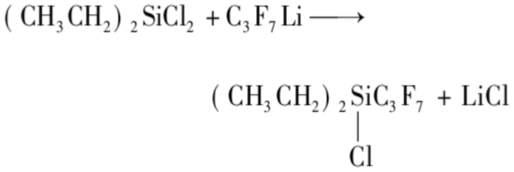


Fig. 8 Synthesis process of fluorosilicone surfactants [3]

2) Organic lithium method

The fluoro-based organic lithium reagent can substitute with the Si-Cl bond to connect the fluorogroup and silicon atoms (as shown in Figure 9); the fluorosilicon surfactant can be obtained by hydrolysis and condensation.



$$(CH_3CH_2)_2SiCl_2 + C_3F_7Li \longrightarrow (CH_3CH_2)_2\underset{\displaystyle Cl}{\underset{|}{Si}}C_3F_7 + LiCl$$

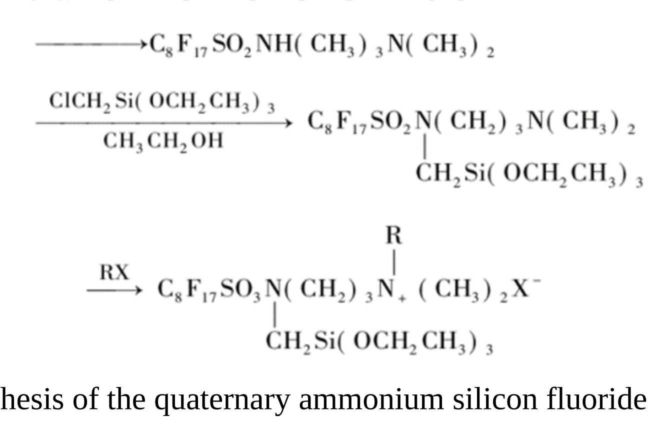
Fig. 9 Synthesis of the fluorosilicon surfactant [3]

3.3.2. The functional group reaction

General functional group reaction methods for synthesizing fluorosilicone surfactants include the quaternary ammonium reaction and epoxy ring-opening reactions.

1) Quaternary ammonium reaction method

The quaternary ammonium reaction is the most common approach for synthesizing fluorosilicone cationic surfactants. This can be achieved either by reacting haloalkanes with amines, such as perfluorotributylamine, with silane or by reacting halogenoalkanes with amine compounds containing silicone [8]. For instance, perfluorosulfonyl fluoride reacts with N, N-dimethyl-1,3-propanediamine in toluene, followed by a reaction with chloromethyltriethoxysilane to obtain N-dimethylamine propyl-N-triethoxysilyl methyl perfluorosulfonamide. This compound can subsequently react with halogenated alkane (RX) to yield a quaternary ammonium silicon fluoride surfactant (as depicted in Figure 10).



$$C_8F_{17}SO_2F + H_2NCH_2CH_2CH_2N(CH_3)_2 \longrightarrow C_8F_{17}SO_2NH(CH_3)_3N(CH_3)_2$$

$$\xrightarrow[CH_3CH_2OH]{ClCH_2Si(OCH_2CH_3)_3} C_8F_{17}SO_2\underset{\displaystyle CH_2Si(OCH_2CH_3)_3}{\underset{|}{N}}(CH_2)_3N(CH_3)_2$$

$$\xrightarrow{RX} C_8F_{17}SO_3\underset{\displaystyle CH_2Si(OCH_2CH_3)_3}{\underset{|}{N}}(CH_2)_3\overset{\displaystyle R}{\overset{|}{N_+}}(CH_3)_2X^-$$

Fig. 10 Synthesis of the quaternary ammonium silicon fluoride surfactant [3]

2) Epoxy-based ring-opening reaction method

The epoxy ring-opening reaction enables the synthesis of various types of fluorosilicone surfactants, including ionic, non-ionic, and amphoteric surfactants [9]. In this process, polysiloxane containing an epoxy group reacts with a secondary amine to form a polysiloxane with a tertiary amine group. This intermediate then reacts with halofluoroalkane to produce the corresponding quaternary ammonium cationic fluorosilicone surfactant. Similarly, polysiloxane with an epoxy group can react with other reagents to yield non-ionic fluorosilicone surfactants (as shown in Figure 11).

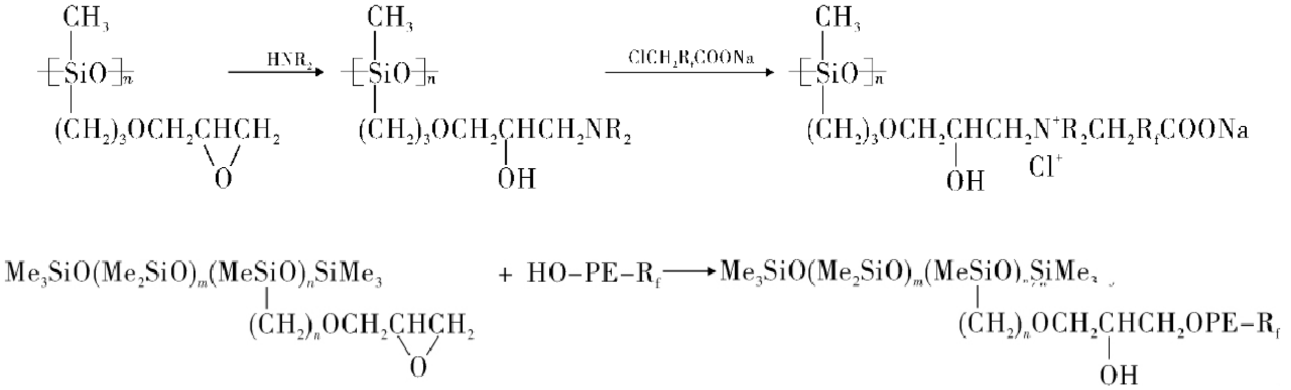
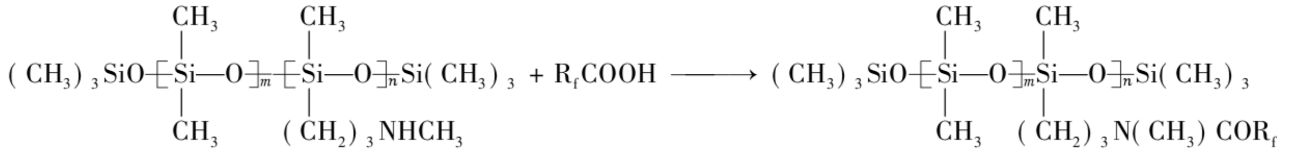


Fig. 11 Synthesis of the non-ionic fluorosilicone surfactants [3]

3) Via reaction perfluoroalkyl carboxylic acid with polysiloxane with a primary amine or secondary amine group [3] (as shown in Figure 12).



$$(CH_3)_3SiO{-}[\underset{CH_3}{\overset{CH_3}{Si}}{-}O]_m{-}[\underset{(CH_2)_3NHCH_3}{\overset{CH_3}{Si}}{-}O]_n{-}Si(CH_3)_3 + R_fCOOH \longrightarrow (CH_3)_3SiO{-}[\underset{CH_3}{\overset{CH_3}{Si}}{-}O]_m{-}[\underset{(CH_2)_3N(CH_3)COR_f}{\overset{CH_3}{Si}}{-}O]_n{-}Si(CH_3)_3$$

Fig. 12 Synthesis of the perfluoroalkyl carboxylic acid with polysiloxane with a primary amine or secondary amine group [3]

Generally, silicon-hydrogen addition and free radical polymerization are the primary methods for synthesizing fluorosilicone surfactants. These methods yield fluorosilicone surfactants with stable Si-C bonds, providing the resulting compounds with excellent stability under varying pH levels and extreme environmental temperatures (both high and low) [10]. The Grignard reagent method is another effective approach for surfactant preparation, known for its high yield, purity, and superior performance, though it requires more stringent reaction conditions. Meanwhile, functional group reactions between silane and fluoroalkane-containing organic compounds, such as quaternary ammonium reactions and epoxy ring-opening reactions, offer relatively easier-to-control conditions, making them an important avenue for producing fluorosilicone surfactants [3].

4. Fluorosilicon Surfactant Applications

4.1. Rubber Industry

Fluorinated and silicone-containing surfactants play a critical role in the rubber industry. The fluorocarbon chains of fluorinated surfactants exhibit both hydrophobic and oleophobic properties, enabling their use as surface modifiers for rubber [1]. This results in the formation of a directional arrangement of perfluoroalkyl chains on the rubber surface, which increases surface smoothness, lowers the coefficient of friction, and ultimately produces cured products with improved adhesion and hardness. Additionally, fluorosurfactants act as antifouling agents by preventing the migration of plasticizers within the elastomer to the surface. The low-friction surface created by the alignment of perfluoroalkyl chains significantly reduces the generation of static electricity during friction, thereby enhancing the antistatic properties of the rubber [11].

Furthermore, silicone-containing surfactants serve as effective coupling agents when used in conjunction with silica fillers in rubber compounds. By modifying silica particles, these surfactants improve interfacial interactions between the filler and the rubber matrix, thereby reducing the formation of inter-particle networks within the silica [11]. As a result, the incorporation of silicone-containing surfactants leads to significant improvements in the abrasion resistance and tensile strength of rubber products.

4.2. Specialty Coatings

The utilization of fluorosilicon surfactants in specialty coatings is based on their inherent properties, including low surface tension, low solubility, excellent dispersibility, durability, and the ability to be effective in low concentrations [12]. As a result, a wide range of varnishes, coatings, and adhesives can be significantly enhanced with minimal quantities of these surfactants. Furthermore, fluorosilicon surfactants help mitigate or eliminate surface tension gradients during the evaporation phase of the coating process, improving the leveling properties of the coating and leading to a more uniform film formation [13].

Fluorosilicone-based coatings demonstrate superior performance compared to conventional coatings, offering attributes such as water repellency, oil repellency, ease of dirt removal, and resistance to chemicals. Additionally, these coatings exhibit resilience under extreme temperatures and possess excellent UV resistance and anti-static properties [13]. These characteristics make fluorosilicone coatings particularly advantageous for use in specialized applications, such as anti-fouling coatings for marine vessels, protective coatings for solar energy devices in arid regions, and various other specialized protective coatings. Their low surface energy and self-cleaning capabilities further contribute to their effectiveness in these applications.

4.3. Metal Lubrication

Fluorosilicon surfactants are also employed in mechanical lubrication, where they form a chemisorbed, molecularly assembled film with high water and oil repellency on the surface of metal substrates. This film, characterized by the presence of -CF_3 groups on its outer surface, significantly reduces the coefficient of friction on the substrate and exhibits superior low-load wear resistance [14]. Notably, when trifluoropropyl silicone oil is used as the base oil in lubricants, the boundary lubrication between steel surfaces is substantially enhanced.

In addition to their lubricating properties, certain fluorosilicon surfactants containing alkenyl groups are utilized as base polymers in solvent-based formulations [15]. These formulations serve as effective strippers, displaying strong adhesion to a wide range of substrates, including paper and plastic films. This versatility further extends the application potential of fluorosilicon surfactants across various industrial sectors.

4.4. Emerging Applications

Fluorosilicon surfactants are demonstrating substantial potential across a variety of emerging fields, particularly in electronics and industrial processes. In the electronics sector, these surfactants are being investigated for their use as chip cleaners due to their low surface energy and superior cleaning properties. Given that the cleanliness of chip surfaces is critical for the operational stability of modern electronic devices, fluorosilicon surfactants are highly effective in removing organic contaminants and microscopic particles without causing damage to the chip material, thereby significantly improving cleaning efficiency [3].

Moreover, in the field of leather treatment, fluorosilicon surfactants are being developed as specialized agents to enhance the waterproofing, anti-fouling, and abrasion-resistant qualities of leather. Their water- and oil-repellent properties enable the formation of a protective film on the leather surface, considerably extending the service life of leather products. This makes them particularly suitable for processing high-end leather goods and specialty leather items [16].

In industrial applications, the use of fluorosilicon surfactants as defoamers is also advancing. Due to their exceptional surface activity, they can swiftly disrupt foam structures on liquid surfaces. These surfactants are particularly effective in addressing defoaming requirements in a range of industrial processes, especially those operating under high-temperature, high-pressure, or corrosive conditions, where they exhibit significant performance advantages [17].

5. Future Trends and Developments

In response to the increasing focus on environmental protection and sustainable development, the synthesis methods of fluorosilicon surfactants are evolving toward more environmentally friendly and efficient approaches. One promising avenue of research lies in the development of novel catalysts that not only reduce the environmental impact of conventional catalysts but also improve the selectivity and conversion efficiency of reactions. Additionally, the exploration of eco-friendly synthesis methodologies, such as solvent-free synthesis and the use of renewable raw materials, represents a pivotal area of future research. These innovations aim to reduce environmental burdens while preserving the high-performance characteristics of the products [18].

The prohibition of fluorinated compounds with side chains longer than six carbons, due to their harmful effects on the environment, organisms, and human health, has spurred efforts to develop biodegradable short chain fluorinated or non-fluorinated alternatives [1]. Recent advancements in short-chain fluorinated polysiloxanes have demonstrated lower toxicity, while providing fabrics with enhanced heat resistance and improved durability in terms of styling and washing.

Structural modifications of fluorosilicon surfactants offer further potential to improve their performance and expand their range of applications. Modifications such as polyether, phosphate ester, sulfonate, quaternization, and polyesterization can enhance specific properties. For example, the modification of phospholipid-type fluorosilicon surfactants has significantly improved their water solubility and detergency, enabling their application in detergents and cleaning agents.

Looking ahead, the potential applications of fluorosilicon surfactants are not limited to traditional industrial sectors. These materials hold promise in renewable energy, aerospace, and advanced manufacturing. In renewable energy, fluorosilicon surfactants could be applied to surface coatings for solar cells and wind power generation equipment, improving weather resistance and self-cleaning capabilities. In the aerospace industry, they may serve as coating materials to enhance corrosion resistance and reduce drag on aircraft surfaces. In advanced manufacturing, fluorosilicon surfactants can be used for lubrication and surface modification of novel materials, improving manufacturing efficiency and product performance.

6. Conclusion

Fluorosilicon surfactants represent an emerging class of surfactants, integrating the unique properties of both fluorinated and silicone-based surfactants. These materials are distinguished by their exceptional chemical stability, weather resistance, corrosion resistance, and hydrophobic and oleophobic characteristics, which have led to their widespread utilization across various industries. The synthesis of conventional fluorosilicon surfactants typically follows two primary methods. The first is the silicon-hydrogen addition method, wherein fluorine- and silicone-containing compounds react with silicon atoms. The second is radical polymerization, which involves the copolymerization of fluorinated substances with silanes containing double bonds via free radical mechanisms.

Fluorosilicon surfactants are extensively applied in fields such as the rubber industry, specialty coatings, and metal lubrication, among others. Further optimization of their molecular structure, including the partial replacement of existing components, offers the potential to enhance their performance in specific applications, thereby expanding their utility in various domains. In conclusion, fluorosilicon surfactants remain an area of active research and demonstrate significant potential for broader application across multiple industries.

Authors Contribution

All the authors contributed equally and their names were listed in alphabetical order.

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