

Carbohydrate-Modified Silicone Surfactants

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Abstract Silicone surfactants have been widely used in our daily life and many industrial fields on the basis of their unusual properties. Only in the past decades has the use of silicone as a hydrophobic building block for the preparation of surfactants become common. The recent trend to combine silicone, polyoxyalkylene and carbohydrate moieties in the same molecule has resulted in a plethora of new compounds with new properties. The generic structure and surface activity of silicone surfactants are reviewed in this article. Especially, the preparation, properties and application of carbohydrate-modified silicone surfactants such as glucosamide-containing and glycoside-containing silicone surfactants are described in detail. In addition, the future development of silicone surfactants is discussed.

Keywords Silicone surfactants · Glucosamide · Glycoside

Introduction

Silicone surfactants are a group of small-molecule and polymeric surfactants. They consist of a permethylated siloxane hydrophobic group (silicone-soluble group) coupled to one or more polar groups (water-soluble group) in the same molecule.

Silicone surfactants exhibit some unusual properties. The siloxane moiety is hydrophobic and oleophobic, which

allows for application in both aqueous and nonaqueous media. They are often liquids even when their molecular weights are rather high. They are usually thermal stability, ultraviolet ray resistant, low toxicity and low surface tension [1–3].

The unique character is closely connected with bonding parameters. The difference of electronegativities of the Si–O bond is 1.76, according to Allred-Rochow. Nevertheless, this polar bond has little influence on the solubility of silicone oils. Evidently, these bonds are shielded by methyl groups that are exposed exclusively at the surface. The Si–O bond length is 0.165 nm compared to 0.140 nm of a C–C bond. The Si–O–Si bond angle of $130 \pm 10^\circ$ is much larger than the corresponding C–O–C bond angle of 110° in dimethyl ether.

Silicone surfactants have been widely used in our daily life and many industrial fields such as polyurethane foam, household, personal care, cosmetics, agricultural adjuvants, textiles and coatings as foam stabilizers, detergents, emulsifying agents, wetting agents and antifoaming/defoaming agents [4–8].

In contrast, carbohydrate surfactants such as *N*-alkyl glucosamides (NAGA) and alkyl polyglucosides (APG) have been known for many years and are particularly interesting because they are (partially) based on renewable resources. In recent years, they have begun to be produced on an industrial scale and their usage has been gradually increasing because of their valuable properties, such as good dermatological compatibility, excellent biodegradability, and the absence of toxic effects.

An increasing number of recent publications have described carbohydrate-modified silicone surfactants. The idea is to combine the extraordinary surface activity and wetting properties of siloxanes with the biodegradability of carbohydrates.

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A summary of the synthesis and the properties of carbohydrate-modified silicone surfactants are given in this review paper.

General Information of Silicone Surfactants

Structure of Silicone Surfactants

Scheme 1 gives a general formula demonstrating organically modified polysiloxanes by substituting various organic groups for methyl moieties.

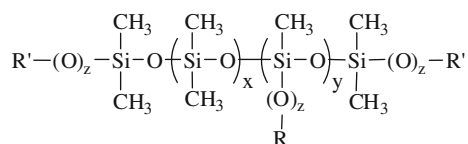
This generic structure describes the variety of organo-modified siloxanes. R and/or R' represent various organic polar groups, which can be aliphatic and/or aromatic; monomers or polymers. They can be attached to a linear, grafted, or branched siloxane backbone via a Si–C bond ($z = 0$) or a Si–O–C bond ($z = 1$). In this way, it is possible to control solubility and compatibility. In general, the organically substituted silicones have relatively short siloxane chain lengths ($x + y = 1\text{--}200$). The organic substituents play a substantial role in the properties of such products.

According to the molecular structure, the grafted polysiloxane is more in the variety of organo-modified siloxanes. The advantage of using grafted copolymers is that they are more resistant to hydrolysis and chemical attack than linear types. According to the linkage of the siloxane backbone and organic groups, the organic groups are attached to the siloxane backbone through a series of hydrolytically stable Si–C bonds by the hydrosilylation reactions or through Si–O–C bonds by a condensation reaction. The latter offers limited resistance to hydrolysis under neutral or slightly basic conditions, but breaks down in strong acidic and basic condition.

The versatility of silicone surfactants comes from the many organic functional groups that can be incorporated into the molecule. Many types of polar groups have been described, but nonionic groups based on polyoxyethylene (PEO) and polyoxypropylene (PPO) have been the most extensively studied.

Surface Activity of Silicone Surfactants

Silicone surfactants have more excellent properties than the organic surfactants. These properties can be attributed to



Scheme 1 The generic structure of organo-modified siloxanes

the strength and flexibility of the Si–O bond, its partial ionic character, and the low interactive forces between the nonpolar methyl groups, characteristics that are directly related to the comparatively long Si–O and Si–C bonds.

Siloxane surfactants significantly reduce the surface tension of the solution at low concentration, indicating that these molecules adsorb strongly at the air/water surface and that they are highly effective aqueous surfactants, reducing the surface tension of water to approximate 21 mN/m.

The low surface tension of silicone surfactants has been attributed to both the preponderance of highly surface active methyl substituent and a flexible polymer backbone. The length of the Si–O and Si–C bonds allows an unusual freedom of rotation, which enables the molecules to adopt the lowest energy configuration at interfaces. By comparison, hydrocarbon surfactants generally have many methylene groups in their hydrophobic portions, which have an intrinsically higher surface energy than methyl groups. The surface tension values of these surfactants suggest that the siloxane portion lies flat on the water surface, exposing the methyl groups to the air.

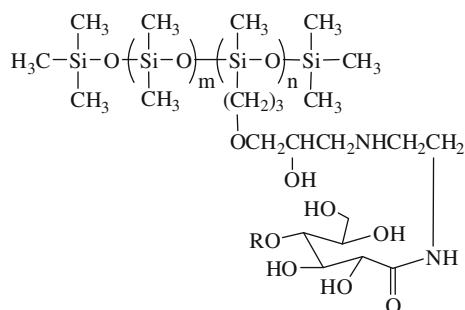
A dramatic change in slope of the surface tension versus log surfactant concentration curves is generally interpreted as the onset of surfactant aggregation into micelles in bulk aqueous solution. Micelle formation by siloxane surfactants in aqueous solution has not been extensively investigated. Most of the studies to date report critical micelle concentrations (CMC) values from surface tension versus log surfactant concentration plots. Although the structure of the micelles formed has been investigated in the past few years, no conclusion has been drawn on the tendency of these surfactants to form well-defined micelles [9–15].

Glucosamide-Modified Silicone Surfactants

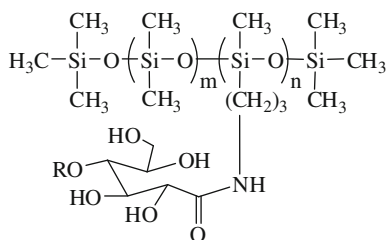
Prepared by Reacting the Aminosilicone with Aldonolactone

Wagner et al. [16, 17] reported a synthetic sequence of reaction of aldonolactones with primary amino functions of polysiloxanes. A series of carbohydrate-modified silicone surfactants (Scheme 2) were synthesized and their properties such as spreading and wetting on a low energy surface (tetrafluoroethylene-hexafluoropropylene copolymer; FEP) were determined.

von Braunmühl et al. [18] synthesized aldonamide siloxanes (Scheme 3) in a broad variety of molecular weight and composition by the hydrosilylation reaction of polymethylhydrosiloxane (PMHS) with *O*-acetylated *N*-allylaldonamides of various sugars using different transition metal complexes as catalyst.



Scheme 2 The carbohydrate-modified silicone surfactants



Scheme 3 The aldonamide siloxanes

Han et al. [19–25] reported the synthesis of a series of glucosamide-modified silicone surfactants such as glucosamide-based trisiloxane surfactants, glucosamide-based trisiloxane surfactants of substitution in the oxyethylene units, and glucosamide-based trisiloxane gemini surfactants (Scheme 4). The properties of these glucosamide-based trisiloxane surfactants were determined by the surface tension, critical aggregation concentration (CAC), the spread abilities on hydrophobic surface (paraffin wax, sterile polystyrene, wheat and cabbage leaf) and the hydrolyzations.

These trisiloxane surfactants significantly reduced the surface tension of the solution to approximately 21 mN/m at low concentration and possessed advantageous spread properties.

Du et al. [26] synthesized carbohydrate-modified siloxane surfactants (Scheme 5) by an analogous method. Their surface activities, adsorption, and aggregation behavior in aqueous solution were investigated by surface tension measurements, dynamic light scattering (DLS) and transmission electron microscopy (TEM).

Prepared by Reacting the Aminosilicone with Glucose

Ma [27] prepared a group of glucosamide gemini surfactants (Scheme 6) by the catalytic hydrogenation reaction between glucose and α,ω -diaminopropylsiloxane in the presence of the Pd/C as a catalyst.

Members of this kind of surfactant reduced the surface tension of water to 25–35 mN/m at concentration levels of 10^{-4} – 10^{-5} mol/L.

Moreover, the shapes of molecular aggregates of gemini surfactants in water were studied by transmission electron microscopy. In the dilute solution, the product displayed unprecedented aggregation behavior with a thread-like micelle-to-vesicle transition. The stability of vesicle was increased as the length of spacer chain grew.

Prepared by Reacting the Epoxy-Silicone with Amino-Sugar

Dietz et al. [28] prepared the siloxane-polyether-sugar ternary copolymers (Scheme 7) by reacting polyether-epoxy-siloxane with *N*-methylglucamine.

Polyether silicones belong to the class of nonionic surfactants which show inverse solubility behavior in water. In other words, they decrease in solubility as the temperature rises. This behavior is defined by what is known as the inverse cloud point. Sugar silicones are likewise nonionic surfactants that exhibit a “normal” solubility behavior. In other words, their solubility in water improves constantly as the temperature climbs. Therefore, through an appropriate balance of polyether groups and sugar groups at the molecular level, it is possible to prepare silicone surfactants with custom-tailored solubility parameters as emulsifiers for use in microemulsions.

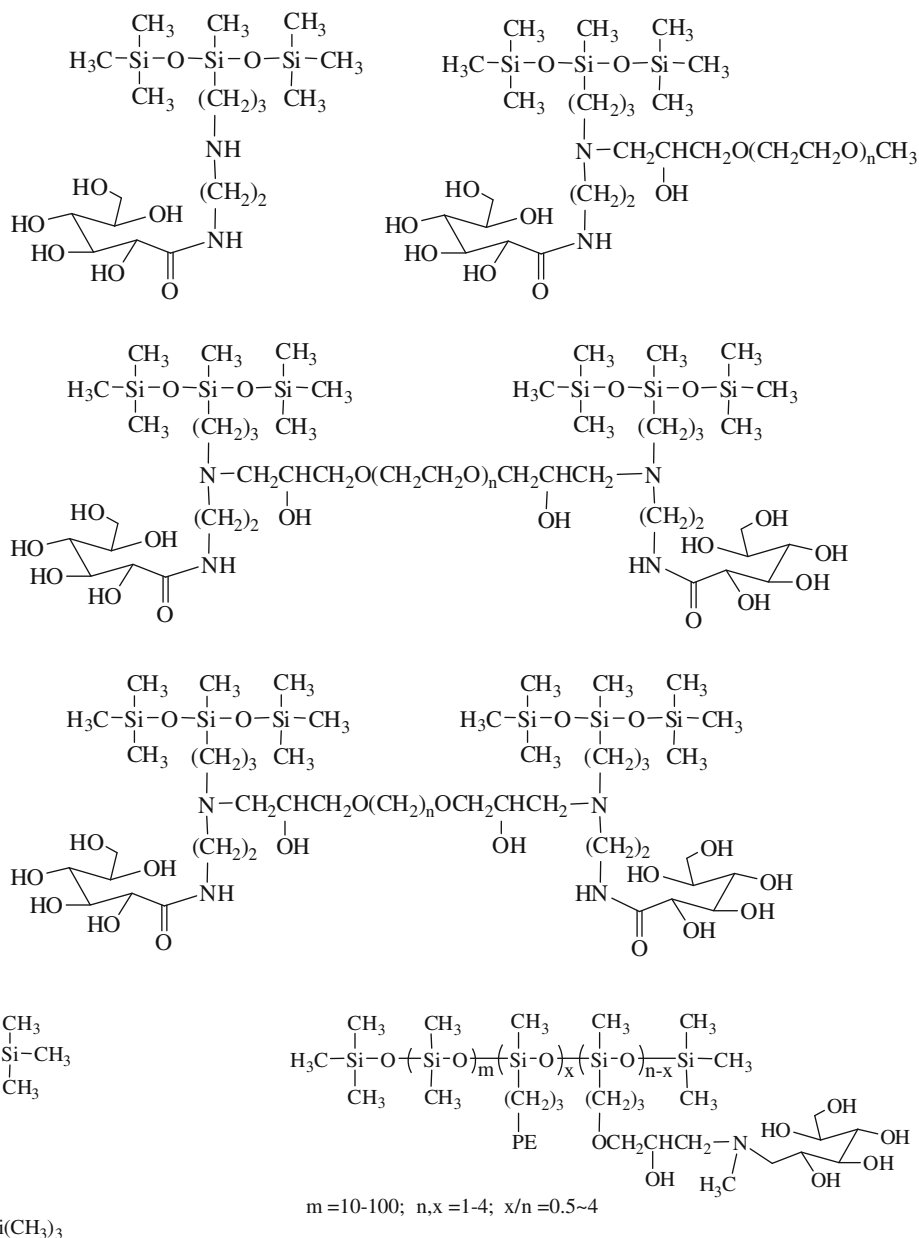
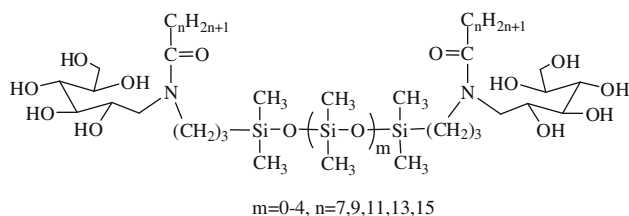
The sugar radicals increase as a proportion of the sum of the sugar and polyether radicals and there is likewise an increase in the cloud point. With a relatively short siloxane chain and an approximately constant number of functional groups per molecule, the cloud point is raised for a comparable proportion of the sugar radicals to the polyether radicals.

Pure sugar siloxanes, despite the large number of OH groups, are of poor solubility in water, probably owing to the formation of aggregates as a consequence of strong hydrogen bonds. The additional polyether radicals in the sugar siloxanes prevent the aggregation of sugar radicals and hence raise the solubility of sugar siloxanes. The sugar-polyether-siloxane features a greatly increased stability in water in comparison with corresponding binary sugar siloxanes.

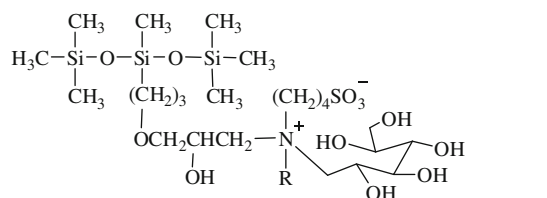
Wagner et al. [29] synthesized sulfobetain amphoteric surfactants (Scheme 8) by means of the reaction of siloxanylpropyl-2,3-epoxypropylether with glucamine or *N*-methylglucamine, respectively, followed by the addition of butanesultone.

The biodegradability, as determined by the OECD test, was 60% higher than that of a common polydimethylsiloxane. But the value of degradability was much lower than it is for hydrocarbon surfactants.

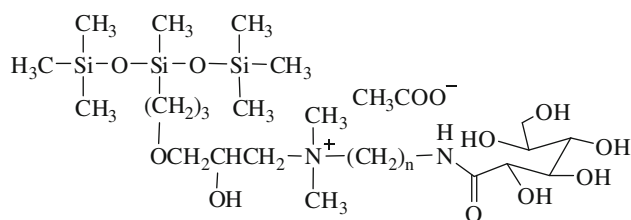
The very high surface activity of this type of siloxane was expressed by the low critical micelle concentration and the maximum surface tension depression. And they were very good emulsifiers.

Scheme 4 The glucosamide-modified trisiloxane surfactants**Scheme 5** The carbohydrate-modified siloxane surfactants**Scheme 6** The glucosamide gemini surfactants

Wagner et al. [30] further reported quaternization of tertiary amino functions containing aminoalkylamides yields polyhydroxylated ammonium salts (Scheme 9).

Scheme 7 The siloxane-polyether-sugar ternary copolymers**Scheme 8** The sulfobetain amphoteric surfactants

The reactions between primary and tertiary amino functions containing polyamines and polyhydroxylated lactones were carried out and yielded the corresponding amides. The quaternary ammonium salts were synthesized by using epoxy-silicone as an alkylating agent. An essential



Scheme 9 The polyhydroxylated ammonium surfactants

precondition for a quantitative alkylation is a sufficiently long alkylene bridge ($n \geq 3$) to insulate the tertiary amino function from electronic (amide structure) influences.

Glycoside-Modified Silicone Surfactants

Prepared by Protective Groups

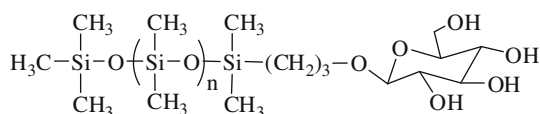
Jonas et al. [31, 32] followed a multistep procedure on the route to carbohydrate-polysiloxane graft copolymers. Peracetylation, glycosidation, hydrosilylation and finally deacetylation yield, mainly β -glycosides of polysiloxanes. Haupt et al. [33] prepared carbohydrate-modified polydimethylsiloxanes (Scheme 10) by an analogous method.

Wagner et al. [16] also synthesized glycoside-modified siloxanes in a four-step sequence which includes peracetylation, glycosidation, deacetylation and hydrosilylation.

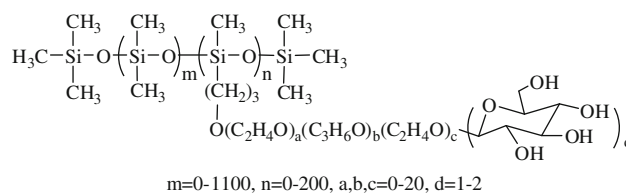
Prepared by Directly Glycosidation

Glycosides bearing siloxanyl moieties have been claimed by Greber [34] and Sejkpa et al. [35]. In both cases, acid catalyzed Fischer glycosidations were described, despite the known tendencies of unprotected reducing carbohydrates to oligomerize and of siloxanes to equilibrate under such conditions.

O'Lenick [36, 37] provided a series of novel silicone containing glycoside polymers (Scheme 11), which were prepared by the reaction of a dimethicone copolyol with a saccharide or saccharide source in the presence of an acid catalyst. They were substantive to skin and hair and possessed outstanding emolency properties when applied to the skin. In addition, unlike the alkyl products previously known, these materials were exceptionally well tolerated by the skin and eye and were essentially non-irritating. The compounds were very mild surface active agents which



Scheme 10 The glycoside modified polydimethylsiloxanes



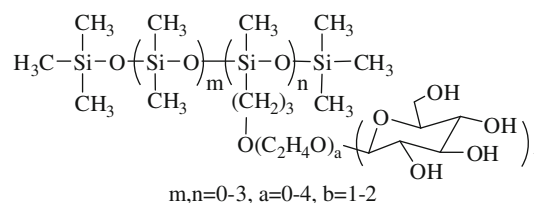
Scheme 11 The glycoside modified dimethicone copolyol

were ideally suited for use in personal care compositions like hair and skin care products.

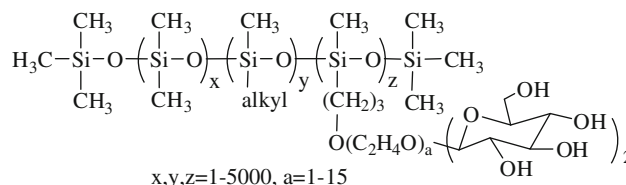
Sejkpa et al. [38] reported that glycoside polysiloxanes (Scheme 12) could be prepared by two various processes. In the first process, the compound whereas synthesized by the hydrosilylation reaction of polymethylhydrosiloxane (PMHS) with allyl polyoxyethylene ether glycosides. The latter was synthesized by the glycosidation reaction of saccharide with allyl polyoxyethylene ether. The excess allyl polyoxyethylene ether can be removed by distillation in the glycosidation. In the second process, the glycoside silicone polymers were prepared by the reaction of a dimethicone copolyol with a saccharide in the presence of an acid catalyst. The excess polyethyleneoxide-modified silicone to lower the polymerization degree of glucoside was not easy to remove. The polysiloxane chain could be equilibrated again by the acid catalyst.

Afriat et al. [39] prepared the siloxane-alkyl-glycoside ternary copolymers (Scheme 13) as a moisturizing agent in a cosmetic or dermatological composition. They were especially effective for treating human skin and the scalp, and were particularly effective for moisturizing the skin and treating dry skin. The composition had long-lasting skin moisturization properties.

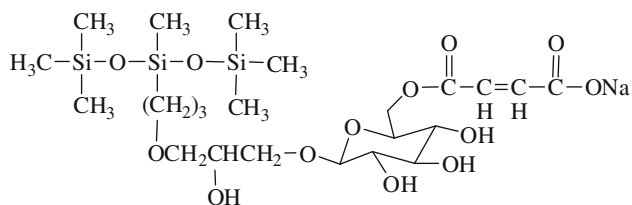
Wagner et al. [40] prepared the anion trisiloxane surfactant (Scheme 14) by the hydrosilylation of allyl glycidyl ether with H-siloxanes followed by the reaction of the



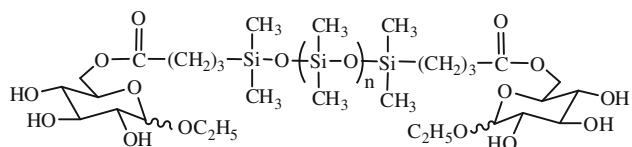
Scheme 12 The glycoside polysiloxanes



Scheme 13 The siloxane-alkyl-glycoside ternary copolymers



Scheme 14 The anion trisiloxane surfactant



Scheme 15 The glycoside-modified siloxane

product with the sodium salt of glucose maleate. The compound was a nearly white solid with good solubility in water and some alcohols.

Using the same reaction path, a number of compounds with polymeric siloxane groups were synthesized. However, they were not soluble in water.

Sahoo et al. [41] synthesized glycoside-modified siloxane (Scheme 15) by esterification between organosilicon carboxylic diacids and a C1-*O*-alkylated sugar by using Novozyme 435 as a catalyst under mild reaction conditions (i.e., low temperature, neutral pH, solventless). Specifically, the acid-functionalized organosilicones reacted with the primary hydroxyl group at the C6 position of α,β -ethyl glucoside during the regioselective esterification. The pure organosilicon-sugar conjugates were prepared in a one-step reaction without protection–deprotection steps and without activation of the acid groups with the integrity of the siloxane bonds.

Further Work

A great variety of silicone derivatives with different structure and functional properties is now available to the formulator. These derivatives permit great formulation latitude and make possible the creation of products which are optimized for specific applications.

The modification of siloxanes with carbohydrate moieties led to a new class of surface active silicone compounds with improved biodegradability. This advance can expand the application of siloxane surfactants into new fields.

Future work on silicone surfactants needs to continue to develop new materials that are able to deliver multiple performance capabilities. A series of surfactants, based on silicone as a hydrophobe and containing other functional groups similar to those in conventional surfactants, have

been and continue to be developed. These surfactants are silicone functional analogs of conventional surfactants.

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