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(54) Title: COATINGS FOR DISEASE CONTROL

(57) Abstract: The present invention provides a method for forming a coating for disease-control on a substrate, the method comprising: providing a compound containing at least two hydrogen atoms attached to one or more nitrogen atoms and a compound containing a cross-linking siloxane precursor group, and applying the compounds to the surface.

COATINGS FOR DISEASE CONTROL

The present invention relates to coatings for disease control.

BACKGROUND TO THE INVENTION

Toxins are biologically-derived molecules that cause disease. They include prions, proteins, polysaccharides, enzymes, nucleic acids and histones. They are, by definition, non-living in contrast to other disease vectors such as pathogens.

In the past, much less research has focused on methods of deactivating toxins compared to killing pathogens because it has been assumed that the most convenient way to render a toxin ineffective is simply to purge the toxin from a system. However, recently it has been found that toxins can be deactivated by materials so that they no longer exhibit their toxic effects. This discovery has partly resulted from an increased interest in toxins caused by events: for example, various protein-based toxins such as ricin and Botulinal toxin have been become more widely available for use as biological weapons.

The greater emphasis in the past on the killing of pathogens such as bacteria, fungi, yeasts and also viruses has caused much effort being made to develop substances that are kill these pathogens. This research has resulted in the design of many substances collectively called 'biocides' that are effective against pathogens. However, little effort has yet been made to develop substances that deactivate toxins. What research that has been done to date has shown that different mechanisms operate in killing pathogens and in deactivating toxins. Therefore, materials that kill pathogens are often not effective against toxins and, as a result, new materials need to be developed that are effective against toxins.

In addition, there is a need to provide more convenient methods of depositing functionalised coatings on the surface of a substrate that produce more resilient coatings than conventional methods, such as coatings that are suitable for disease control.

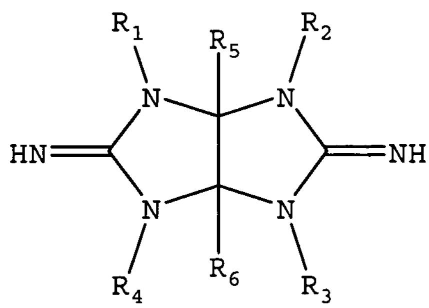
SUMMARY OF INVENTION

The present invention aims to address at least some of the problems of the prior art. Accordingly, the present invention provides in a first aspect a method for forming a coating on a substrate for deactivation of toxins, the method comprising: providing a compound containing a glycoluril functional group and a siloxane monolayer precursor group, applying the compound to the surface, and exposing the surface to microwave electromagnetic radiation.

In a second aspect, the present invention provides a method for forming a coating for deactivation of toxins on a substrate, the method comprising: providing a compound containing a functional group containing at least two hydrogen atoms attached to one or more nitrogen atoms and a compound containing a cross-linking siloxane precursor group and applying the compounds to the surface.

In a third aspect, the present invention provides substrate having a coating attached to, and / or organised into an array on a surface by being treated by the method of the first or second aspects.

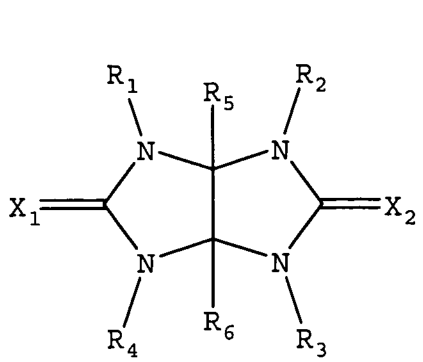
In a fourth aspect, the present invention provides a compound having the structure:



(6)

wherein: R_1, R_2, R_3 and R_4, R_5 and R_6 are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl, a group containing a siloxane monolayer precursor, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl), and at least one of R_1, R_2, R_3, R_4, R_5 and R_6 is a group containing a siloxane monolayer precursor.

In a fifth aspect, the present invention provides a compound having the structure:



wherein: X_1 and X_2 are independently-selected heteroatoms, optionally having one or more pendant independently-selected alkyl and / or independently-selected heteroalkyl groups and / or hydrogen, R_1, R_2, R_3 and R_4, R_5 and R_6 are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl), and at least one of R_1, R_2, R_3, R_4, R_5 and R_6 is a group containing a vinyl group, an imide, an acrylate, an alkene, an epoxide or an alkyl halide.

In a sixth aspect, the present invention provides a method for forming a coating for deactivation of toxins on a substrate, the method comprising: providing the compound of the fifth aspect, applying the compound to the surface, and exposing the surface to microwave electromagnetic radiation.

In a seventh aspect, the present invention provides a substrate having a compound containing a glycoluril functional group attached to, and / or organised into an array on the surface by the method as defined in the sixth aspect.

The present invention also provides the use of a coating formed from a compound containing a glycoluril functional group and a siloxane monolayer precursor group in the deactivation of toxins. The present invention also provides the use of a coating formed from by the method as defined in the first aspect in the deactivation of toxins.

Finally, the present invention provides a method for forming a coating on a substrate, the method comprising: providing a compound containing a glycoluril functional group and a compound containing a cross-linking siloxane precursor, and applying the compound to the surface.

DETAILED DESCRIPTION OF THE INVENTION

The present invention will now be further described. In the following passages different aspects of the invention are defined in more detail. Each aspect so defined may be combined with any other aspect or aspects unless clearly indicated to the contrary. In particular, any feature indicated as being preferred or advantageous may be combined with any other feature or features indicated as being preferred or advantageous.

The inventor of the present invention has found a class of molecules that is effective against toxins when attached to a surface in a particular way. This molecule includes a glycoluril functional group. The molecule further includes a siloxane monolayer precursor group so that the glycoluril functional group is attached to a surface and / or organised into an array at the surface by reaction of the surface with the siloxane monolayer precursor.

Conventionally, this molecule would be attached to the surface by heat treatment as in US 6969769. However, the inventor has found that this method of attachment of the molecule to the surface does not result in a surface that is effective against toxins. Instead, the inventor has found that it is necessary to attach this molecule and other molecules containing a N-halogen bond to a surface under the irradiation of microwaves in order for them to form a surface that is effective against toxins.

Accordingly, in a first aspect, the present invention provides a method for attaching compounds containing both a glycoluril functional group and a siloxane monolayer precursor group to a surface by exposing the surface to the compound while being treated with microwaves.

The first aspect also more broadly provides a process for deactivating toxins, the process comprising: exposing a toxin to a coating deposited on a substrate, wherein the coating comprises an N-halogen group. Preferably, the N-halogen group is contained in a compound containing a functional group selected from the group consisting of hydantoin, imidazolidinone, glycoluril, iscyanurate and triazinedione. Preferably, the halogen appends one of the nitrogen atoms contained in a of hydantoin, imidazolidinone, glycoluril, iscyanurate or triazinedione group. In particular, the inventor has found a way in which a surface may be provided having a density of N-halogen bonds to be effective against toxins, namely by deposition of the compounds containing the N-halogen group(s) or precursors to the N-halogen groups(s) under the treatment of microwaves. Preferably, the coating is deposited by the method of the second aspect of the invention in combination with microwave irradiation.

As used herein, the term *"microwaves"* preferably refers to electromagnetic radiation having a frequency from 0.3 to 30 GHz. Preferably, the microwaves have a frequency of 0.3 to 10 GHz, more preferably from 1 to 3 GHz. The microwaves are actively applied to the substrate by, for example, a microwave power source. The microwaves may be produced using a power rating of 650 Watts or less, for example 65 to 650 Watts, such as 135 to 400 Watts.

As used herein, the term *"siloxane monolayer precursor"* refers to a group that is able to form a siloxane monolayer at the surface of a substrate. Siloxane monolayer precursors are well-known to the person skilled in the art.

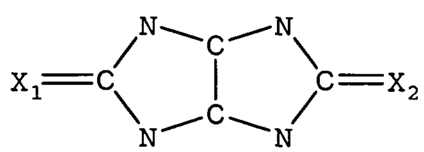
Typically, the surfaces for use in the present invention contain a number of nucleophilic sites on their surface, so reaction of the siloxane monolayer precursor with a surface typically involves the nucleophilic displacement of a leaving group attached to the silicon by one of the nucleophilic sites on the surface of the substrate. At the same time, the siloxane monolayer precursor may also react with other siloxane monolayer precursor molecules in a cross-linking reaction to from the Si-O-Si (siloxane) functional group to form an organised array of molecules. In addition, the term *"siloxane monolayer precursor"* includes within its scope pre-formed siloxane groups, such as siloxane polymers. This overall process is known as the self-assembly of a monolayer at the surface.

It should be noted that the term *"siloxane monolayer precursor"* does not require the formation of a complete monolayer over the surface of a substrate. In fact, the formation of a complete surface-covering monolayer involves the careful control of reaction conditions and selection of precursors. Rather, the term simply requires that some of the siloxane monolayer precursor groups contain groups such as leaving groups that can be displaced by nucleophiles that can react with the surface of a substrate. If the siloxane monolayer precursor does not already contain siloxane bonds, the precursor may also be able to self-react with other siloxane monolayer precursor molecules (self-reaction usually occurs after the displacement of a leaving group with, for example, water, followed by reaction of the newly formed Si-OH with a second siloxane monolayer precursor). In addition, the term does not exclude precursor molecules that form multi-layers rather than single monolayers.

As previously noted, examples of siloxane monolayer precursors are well-known to the person skilled in the art. Siloxane monolayer precursors include compounds containing silicon-X_3 functional groups, wherein X_3 is a leaving group. X_3 may be, for example, a halogen, O-alkyl, O-heteroalkyl, OH, NH_2, NH-alkyl, NH-heteroalkyl, N(alkyl)(alkyl), N(alkyl)(heteroalkyl) or N(heteroalkyl)(heteroalkyl). Siloxane monolayer precursors also include pre-formed siloxane polymers themselves.

For example, the siloxane monolayer precursor may be selected from a siloxane compound, a silanol compound, a silyl ether compound, a silanolate compound, a halosilane compound, a silatrane compound and a silazane compound.

As used herein, the term *"glycoluril functional group"* refers to a compound containing the following chemical structure:



(1)

In this structure, X_1 and X_2 are independently-selected optionally-substituted heteroatoms (i.e. atoms other than carbon). The heteroatoms may be substituted with one or more independently-selected alkyl and / or heteroalkyl groups (as defined below) and / or hydrogen as appropriate. For example, X_1 and / or X_2 may be oxygen, nitrogen or sulphur. If either or both of X_1 or X_2 is nitrogen or another atom having a valency of three or more, X_1 and / or X_2 is substituted with further substituents. For example, X_1 and / or X_2 may have the chemical formula NR_7. In a preferred embodiment, R_7 (or any other substituent(s)) is hydrogen. Alternatively, R_7 (or any other substituent(s) on any heteroatom) may be OH, O-alkyl, O-heteroalkyl, alkyl or heteroalkyl. It is to be noted that the carbon-carbon bond shown in the above structure may be a single bond or a double bond.

The term *"alkyl"* refers to a group containing carbon and hydrogen. An alkyl group contain any number of carbon atoms. Preferably, the alkyl group contains 1 to 25 carbon atoms. More preferably, the alkyl group contains 1 to 10 carbon atoms, for example 1 to 6 carbon atoms. The alkyl group may itself be unsubstituted (i.e. contain only carbon and hydrogen) or, alternatively, it may be substituted with heteroatom-containing substituents. The alkyl group may straight-chained or it may be branched or it may cyclic, or combinations thereof. The alkyl group may be saturated, partially or completely unsaturated or aromatic. For example, the alkyl group may comprise one or more alkene and / or alkyne functional groups. Equally, the alkyl group may be or may comprise an aryl group. The term *"aryl"* group refers to a group comprising one or more aromatic cycles. The cycle is made from carbon atoms.

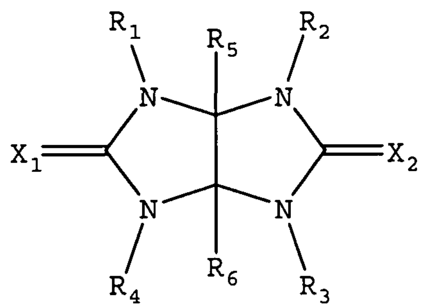
Examples of saturated unsubstituted alkyl groups having 1 to 6 carbon atoms include methyl, ethyl, *n*-propyl, *sec*-propyl, cyclopropyl, *n*-butyl, *sec*-butyl, *tert*-butyl, cyclobutyl, pentyl (branched or unbranched) and hexyl (branched or unbranched). Examples of saturated unsubstituted alkyl groups having 1 to 6 carbon atoms further include cyclic carbon compounds, for example a cyclopropyl group, a cylcobutyl group, a cyclopentyl group and a cyclohexyl group. If the alkyl group is substituted, it may be partially substituted or completely substituted with one or more independently selected heteroatoms or groups of heteroatoms. Examples of heteroatom substituents include the halogens, including fluorine, chlorine, bromine or iodine, -OH, =O, $-NH_2$, =NH, -NHOH, =NOH, $-OPO(OH)_2$, -SH, =S or $-SO_2OH$. If the alkyl group is substituted with =O, the alkyl group may comprise an aldehyde, a ketone, a carboxylic acid or an amide.. An example of an aryl substituent is a phenyl group.

For example, alkyl may be haloalkyl, such as a haloalkyl in which one or more halo groups is located at the distal end of the alkyl chain from the silicon. The haloalkyl is preferably chloroalkyl.

The term *"heteroalkyl"* group refers to a first alkyl group substituted with one or more independently-selected heteroatoms or groups of heteroatoms, which itself is substituted with one or more independently-selected groups containing one or more carbon atoms. As such, a heteroalkyl can be represented by the generic formula R_8 - Y - R_9, where R_8 is an alkyl group, Y is one or more heteroatoms and R_9 contains one or more carbon atoms and optionally one or more heteroatoms and connects to Y through a carbon atom (e.g. an alkyl group). Examples of heteroatoms in heteroalkyl groups include nitrogen, oxygen, phosphorus and sulfur. In one embodiment, Y as defined above is oxygen. In another embodiment, Y is a nitrogen atom. It is also to be noted that R_8 and R_9 may be joined to one another so as to form a cyclic group containing one or more heteroatoms.

A heteroalkyl group contain any number of carbon atoms. Preferably, a heteroalkyl group contains a total of 1 to 25 carbon atoms. In one embodiment, the heteroalkyl group contains a total of 1 to 10 carbon atoms, for example 1 to 6 carbon atoms. The heteroalkyl group may itself be unsubstituted (i.e. contain only carbon, hydrogen and the heteroatom or groups of heteroatom contained in the backbone of the heteroalkyl group) or, alternatively, it may be substituted with heteroatom-containing substituents. The heteroalkyl group may straight-chained or it may be branched or it may cyclic, or combinations thereof. The heteroalkyl group may be saturated, partially or completely unsaturated or aromatic. The heteroalkyl group may be or may comprise cyclic groups containing a heteroatom. As described above for alkyl groups, the heteroalkyl group may be unsubstituted or substituted with one or more hetero-atoms or group of hetero-atoms. Alternatively or in addition, the heteroalkyl group may be substituted with one or more heteroatoms or groups of heteroatoms that are themselves substituted with one or more independently selected alkyl groups. The heteroalkyl group may also be or may comprise a heteroaryl group. The term *"heteroaryl"* refers to group comprising one or more aromatic cycles. The cycle is made from carbon atoms and heteroatoms. The one or more heteroatoms are independently-selected from, for example, nitrogen, oxygen, phosphorus and sulphur.

Preferably, the first aspect of the present invention uses a glycoluril compound having the following structure:

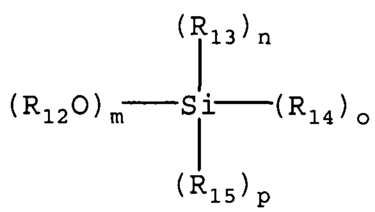


(2)

In formula (2), X_1 and X_2 are independently-selected optionally-substituted heteroatoms (i.e. atoms other than carbon). For example, X_1 and / or X_2 may be oxygen, nitrogen or sulphur. If either X_1 or X_2 is nitrogen, the nitrogen is substituted with a third substituent, i.e. X_1 and / or X_2 have the chemical formula NR_7. In a preferred embodiment, this third substituent (R_7) is hydrogen. Alternatively, R_7 (or any optional substituent) may for example be OH, O-alkyl, O-heteroalkyl, alkyl or heteroalkyl.

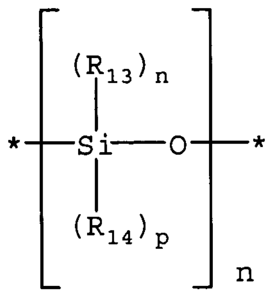
In formula (2), R_1, R_2, R_3 and R_4 are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl and a siloxane monolayer precursor-containing group. Preferably, at least one of R_1, R_2, R_3 and R_4 is hydrogen, a halogen (preferably chlorine and / or bromine) or a protecting group for hydrogen (protecting groups are well-known in the art: see, for example, the book *"Protective Groups in Organic Synthesis"* by Greene *et al.*). R_5 and R_6 are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl and a siloxane monolayer precursor-containing group, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl). At least one of R_1, R_2, R_3, R_4, R_5 and R_6 is a siloxane monolayer precursor-containing group.

The at least one siloxane monolayer precursor-containing group may have the following chemical structure:



(3)

or a polymer have repeating units of formula (4), which may be terminated by hydrogen, hydroxyl, an alkyl or an amine group at one or both ends of the polymer chain:



(4)

wherein:

R_{12} is hydrogen or an alkyl group or a heteroalkyl group, preferably a C_1 to C_6 alkyl, more preferably, a C_1 or C_2 alkyl, such as methyl or ethyl, and m is 1 to 4, preferably 3;

R_{13}, R_{14} and R_{15} are each independently selected from alkyl, aminoalkyl, heteroalkyl and aminoheteroalkyl,

in formula (3), n, o and p are each 0 to 3, providing that $m + n + o + p = 4$, ($m + n + o + p$ can also equal up to 6),

in formula (4), n and p are each 0, 1 or 2, providing that $n + p = 2$, and

at least one of R_{13}, R_{14} and R_{15} contains a glycoluril functional group.

Other leaving groups may replace OR_{12} in formula (3) above, for example one or more halogens (e.g. Cl). Leaving groups may also replace R_{13} or R_{14} in formula (4) above.

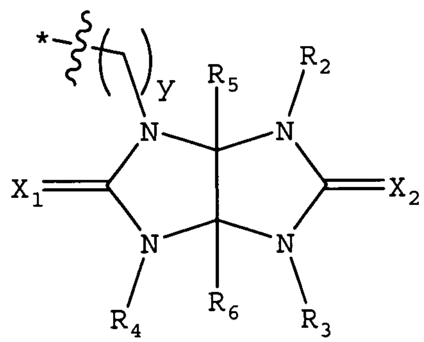
Examples of alkyl and heteroalkyl groups for use in the present invention include groups containing glycidoxy groups, amino groups, acrylates and groups containing alkenes.

The polymer defined above preferably includes electron donor groups on at least some of its monomers. These electron donor groups may be substituents on R_{13} and / or R_{14} in formula (4) above or on on R_{13} and / or R_{14} and / or R_{15} in formula (3) above. Electron donor groups include, but are not limited to, hydroxyl, amine, sulfhydryl and carboxyl. X, the number of repeating units in the polymer, may be any appropriate number. X may be 2 to 10000, preferably 2 to 1000.

Preferably, R_{12}, R_{13}, R_{14} and / or R_{15} are substituted with one or more halogens. Preferably, the halogens are located at the distal end of the group(s) from the silicon. Preferably, the halogen is chlorine.

Preferably, the siloxane monolayer precursor-containing compound is a compound of formula (3), wherein m is 3, n is 1 and o and p are both 0, R_{12} is hydrogen, methyl or ethyl.

Preferably, at least one of R_{13}, R_{14} and / or R_{15} is of the formula (5):



(5)

wherein y is 1 to 5, preferably 3.

Preferably R_5 and / or R_6 is / are a UV-stabilizing group.

In order to deposit the siloxane monolayer precursor-containing compound on a surface, a solution or suspension of the silicon-containing compounds may be contacted with the surface. The solution and / or suspension preferably comprises a polar solvent, more preferably acetone and / or alcohol, preferably both. The alcohol preferably comprises methanol and / or ethanol. Alternatively, the siloxane monolayer precursor-containing compound may be solvent-free, i.e. not in the form of a solution or suspension, for example in the form of a liquid or gas (but preferably not a plasma).

The surface of the substrate is preferably a material having nucleophilic sites on its surface. The nucleophilic sites may comprise one or more nucleophilic groups containing one or more of O, S and N. For example, the nucleophilic groups may be oxygen-containing, nitrogen-containing and / or sulfur-containing, for example selected from OH, SH and NH_2. The substrate may comprise a fabric material. It has been found that the nucleophilic groups bind to the silicon atoms of the siloxane monolayer precursor-containing compounds on contact and with exposure to microwaves. This reaction normally occurs within seconds, as opposed to hours for conventional methods, such as merely heating.

In practice, in order to reduce the possible degradation of delicate siloxane monolayer precursor-containing compounds and / or delicate substrates, one or more of the following may be used: irradiation at a reduced power level, for example microwaves produced at a power rating of 400 Watts or less, preferably 135 Watts or less, or subjecting the substrate and siloxane monolayer precursor-containing compounds to *microwave irradiation and relaxation* (i.e. *no microwave irradiation*) *in alternating* intervals: for example a period of irradiation of preferably 5 to 30 seconds, more preferably 10 to 20 seconds, most preferably about 15 seconds, followed by a period of relaxation of preferably 2 to 30 seconds, more preferably 5 to 15 seconds, most preferably about 10 seconds, and optionally repeating this process as often as required. It has been found that, for many compounds containing an Si-O moiety, this is more sensitive to microwave radiation than other 'delicate' functionalities and therefore cleavage of the Si-O bond may be achieved without degradation of the other functionalities.

The microwaves can be directed at particular portions of the substrate and therefore allow for regioselective attachment and / or arrangement of the silcon-substituted compounds and for reactions that can be initiated that would not be possible using traditional methods.

The substrate may comprise a natural material. The material may be a cloth material. The material may comprise one or more materials selected from cotton, wool and leather. The material may be woven or non-woven. The material may comprise fibres of natural and / or synthetic material. The synthetic material may comprise a woven or nonwoven fabric material to include, but limited to, fabrics wherein the material comprises one or more of cotton, polyester, nylon, wool, leather, rayon, polyethylene, polyvinylchloride, polyvinylalcohol, polyvinylamine and polyurea.

The substrate may be in the form of particles. The particles may have a diameter of 10 nm to 1 mm, preferably 100 to 1000 nm.

The substrate may comprise a metal oxide. The metal oxide may be selected from one or more of aluminium oxide, titanium dioxide, magnesium oxide, calcium oxide, silicon dioxide and zinc oxide.

The substrate may comprise a natural mineral. The substrate may comprise one or more materials selected from kaolinite, barasym, silica, montmorillonite, vermiculite, bohemite and quartz.

The substrate may be porous. The substrate may comprise a molecular sieve. The substrate may comprise a zeolite.

The substrate may comprise a polymer. The polymer may be in the form of a porous matrix. The substrate may comprise a plastic material. The substrate may comprise polyurethane and / or nylon, plyester, nylon, rayon, polyethylene, polyvinylchloride, polyvinylalcohol, polyvinylamine and polyurea.

The substrate may comprise a carbohydrate.

An alcohol may be present during deposition of the coating. In particular, the substrate may comprise an alcohol. The substrate may have an alcohol on its surface. The alcohol may comprise a diol, which may be a vicinal diol, or a triol. The alcohol may be selected from one or more of an alkyl diol, preferably a C_2 to C_{25} alkyl diol, an alkyl trio, preferably a C_3 to C_{25} alkyl triol and a phenyl diol, preferably a vicinal phenyl diol. Each hydroxyl group in the triol is preferably vicinal to one of the other hydroxyl groups. The alcohol may be selected from catechol, ethylene glycol or glycerol.

The substrate may comprise a silicon dioxide based material, such as glass, silicon dioxide, sand and silica.

In order to give the glycoluril functional group of the present invention its disease-prevention properties, the glycoluril group should have at least one of its nitrogen substituted with a halogen, for example chlorine or bromine (i.e. at least one or R_1, R_2, R_3 and R_4 should be a halogen). The halogen may be introduced at any stage. For example, it may be introduced prior to the deposition of the coating on the substrate. Alternatively, it may be introduced after the deposition of the coating on the substrate. For example, the glycoluril group may be halogenated with an oxidative halogen compound, such as a hypochlorite, for example aqueous sodium hypochlorite. The concentration of chlorine at the surface of the substrate may be measured by iodometry, for example in ppm defining the surface molar concentration of the chlorine.

Following treatment of the substrate with the halogenating agent, the substrate may be dried. The substrate may be dried by exposing it to a temperature of 20°C or more, preferably 30°C or more, more preferably 35°C for a period including, but not limited to, 1 hour or more, preferably 4 hours or more.

In a second aspect, the present invention provides a convenient method for attaching a functional compound such as those containing disease-preventing functional groups to a surface. This method involves providing a compound containing at least one nucleophilic group, such as a compound containing at least two hydrogen atoms attached to one or more nitrogen atoms, and a separate compound containing a cross-linking siloxane precursor group. (Thus , the compound may contain for example either NH_2 or NH_3^+ or two or more nitrogens of the formula NH_x, where x is 1 to 3 and where x is independently selected for each amine functional group. Other groups are of course attached to the NH_x as so defined). The compounds are applied to the surface of the substrate at the same time as one another. Preferably, the surface is then exposed to microwave electromagnetic radiation (preferably substantially at the same time as applying the compounds to the surface of the substrate).

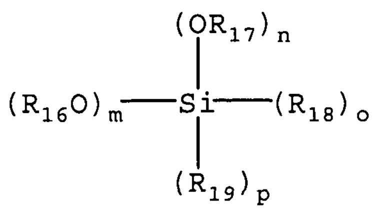
As used herein, the term *"compound containing a siloxane precursor cross-linking group"* refers to a compound that contains one or more silicon atoms that are in total capable of reaction with at least two nucleophiles. The compound may alternatively be referred to as a silicon-containing compound having two or more leaving groups. In use, the compound is capable of reacting with the surface (whose preferable characteristics are the same as the first aspect), with the glycoluril compound itself and optionally with itself (to form a siloxane polymer).

In total, the siloxane precursor cross-linking compound is able to undergo at least two reactions in which nucleophiles displace a leaving group at one or more silicon atom. In other words, the compound preferably contains a total of at least two leaving groups. These may append either the same silicon atom or, if the compound contains more than one silicon atoms, these may append different silicon atoms. With at least two leaving groups, the compound is able to both react with a surface and also with a glycoluril-containing compound. As such, its function is to cross-link the surface to the glycoluril-containing compound.

The leaving groups appended to the silicon atom(s) in this second aspect are the same as in the first aspect. The terms *"leaving group"* and *"nucleophile"* are well known in the art (see, for example, "Guidebook to Mechanism in Organic Chemistry" (1986) by Peter Sykes). Thus, *"leaving group"* refers to a moiety that is easily displaced and replaced by a nucleophile. Common leaving groups include moieties that are relatively stable once displaced and may include be moieties that are stabilised in the presence of acidic or basic condition. Common moieties are of the formula RO^-, wherein R is hydrogen or an alkyl, preferably a C1 to C6 alkyl, more preferably a C1 or a C2 alkyl, such as methyl or ethyl. Other leaving groups include amines, alkylamines (C1 to C6 preferably), carboxylates (C1 to C6 preferably), alkylamides (C1 to C6 preferably), halides, azides and thiocyanates. In the above alkyl group chain length ranges, C1 and C2 alkyl groups are preferred. In particular, the leaving groups may be silicon-X_3 functional groups, wherein X_3 is hydrogen, a halogen, O-alkyl, O-heteroalkyl, OH, NH_2, NH-alkyl, NH-heteroalkyl, N(alkyl)(alkyl), N(alkyl)(heteroalkyl) or N(heteroalkyl)(heteroalkyl). Alternatively or additionally, siloxane polymers themselves may be suitable.

Preferably, the siloxane precursor cross-linking compound has a total of at least three leaving groups appending one or more silicon atom(s). This is so that they may undergo three separate reactions, namely reaction with a surface, reaction with the glycoluril-containing compound and self-reaction to form a organised array. For example, the siloxane precursor cross-linking compound may comprise at least four leaving groups appending one or more silicon atoms.

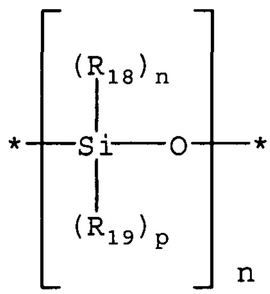
Examples of suitable siloxane precursor cross-linking compounds include compounds having the chemical structure:



(6)

wherein in formula (6) m and n are each 0 to 4, o and p are each 0 to 2, providing that m + n + o + p = 4, and m + n is at least 2, preferably 3, for example 4, (m + n + o + p can also equal up to 6),

or a polymer have repeating units of the following formula, which may be terminated by hydrogen, hydroxyl, an alkyl or an amine group at one or both ends of the polymer chain:



(7)

wherein:

R_{16} and R_{17} are independently selected from hydrogen, an alkyl group or a heteroalkyl group, preferably a C_1 to C_6 alkyl, more preferably, a C_1 or C_2 alkyl, such as methyl or ethyl;

R_{18} and R_{19} are each independently selected from alkyl, aminoalkyl, heteroalkyl and aminoheteroalkyl,

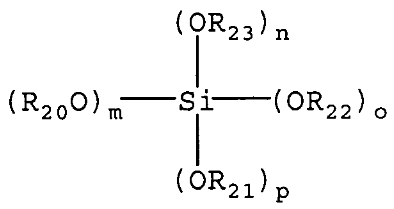
in formula (7), n and p are each 0, 1 or 2, providing that n + p = 2.

Other leaving groups may replace OR_{16} or OR_{17} in formula (6) above, for example one or more halogens (e.g. Cl). Leaving groups may also replace R_{18} or R_{19} in formula (7) above.

The polymer defined above preferably includes electron donor groups on at least some of its monomers. These electron donor groups may be substituents on R_{18} and / or R_{19} in formulas (6) and (7) above. Electron donor groups include, but are not limited to, hydroxyl, amine, sulfhydryl and carboxyl. X, the number of repeating units in the polymer, may be any appropriate number. X may be 2 to 10000, preferably 2 to 1000.

Preferably, R_{18} and / or R_{19} are substituted with one or more halogens. Preferably, the halogens are located at the distal end of the group(s) from the silicon. Preferably, the halogen is chlorine.

Preferably, the siloxane precursor cross-linking compound is a compound having the following structure:



(8)

where R_{20}, R_{21}, R_{22} and R_{23} are independently selected from hydrogen, an alkyl group or a heteroalkyl group, preferably a C_1 to C_6 alkyl, more preferably, a C_1 or C_2 alkyl, such as methyl or ethyl. In one embodiment, R_{16}, R_{17}, R_{18} and R_{19} are the same and may preferably be chosen to be methyl or ethyl. In particular, these compounds may be particularly effective in forming a resilient coating having functionality.

For example, the siloxane precursor cross-linking compound may be selected from orthosilicic acid, a tetraalkoxysilane, preferably tetramethoxysilane or tetraethoxysilane, a tetraacyloxysilane, preferably tetraformyloxysilane or tetraacetoxysilane, tetraminosilane, or a tetra(alkylamino)silane. Preferably, tetraethylorthosilicate ($Si(OCH_2CH_3)_4$) is used because it has been found that this compound is effective in forming a functionalised surface coating.

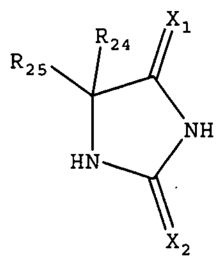
In order to react with the siloxane precursor cross-linking compound, a functional compound may be provided that contains at least two hydrogen atoms attached to one or more nitrogen atoms.

It is to be noted that the *"compound containing at least two hydrogen atoms attached to one or more nitrogen atoms"* includes within its scope compounds in which one or more of the hydrogens are replaced by a protecting group so long as the compound contains at least one hydrogen attached to a nitrogen atom. In this case, after reaction with the cross-linking siloxane precursor group, the protecting group is de-protected exposing another hydrogen attached to a nitrogen atom. Alternatively, the hydrogen may be being protected by being replaced by a halogen (for example chlorine or bromine), in which case there is no need to de-protect the nitrogen because it already has some disease-preventing properties. Preferably, the compound does not contain a silicon atom.

In this aspect, the known disease-control properties of a N-halogen group may be taken advantage of. While the functional compound may be selected and certain processing conditions (e.g. microwave irradiation during / shortly after exposure of the surface to the functional compound and the siloxane precursor) may be used to render a surface effective against toxins, the N-halogen group is known also to be effective against pathogens. Accordingly, this aspect is not restricted to the specifically-selected groups of the first aspect of the invention but is much more widely applicable to disease-control coatings and other functional coatings.

The *"compound containing at least two hydrogen atoms attached to one or more nitrogen atoms"* may be cyclic or acyclic. Preferably, it is cyclic. Preferably, the compound contains a functional group selected from the group consisting of hydantoin, imidazolidinone, glycoluril, isocyanurate and triazinedione.

The hydantoin functional group preferably has the following chemical formula:

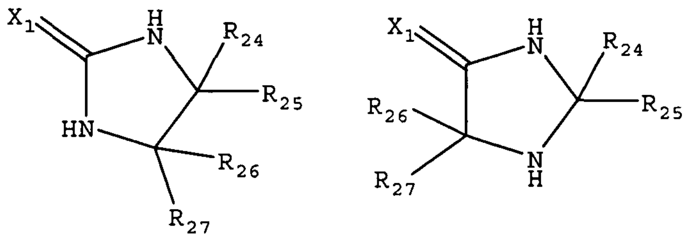


and derivatives thereof in which one of the NHs shown in the above formula is either protected with a protecting group or is replaced by N-Halogen (preferably N-Cl or N-Br). Protecting groups are well-known in the art (see, for example, the book *"Protective Groups in Organic Synthesis"* by Greene *et al.*).

In this formula, X_1 and X_2 are independently-selected optionally-substituted heteroatoms (i.e. atoms other than carbon). For example, X_1 and / or X_2 may be oxygen, nitrogen or sulphur. If either X_1 or X_2 is nitrogen, the nitrogen is substituted with a third substituent, i.e. X_1 and / or X_2 have the chemical formula NR_7. In a preferred embodiment, this third substituent (R_7) is hydrogen. Alternatively, R_7 (or any other optional substituent) may be OH, O-alkyl, O-heteroalkyl, alkyl or heteroalkyl.

In this formula, R_{24} and R_{25} are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl and a siloxane monolayer precursor-containing group, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl).

The imidazlidinone functional group preferably has the following chemical formula (it can have two forms):

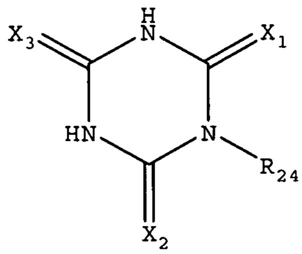


and derivatives thereof in which one of the NHs shown in the above formulae is either protected with a protecting group or is replaced by N-Halogen (preferably N-Cl or N-Br). Protecting groups are well-known in the art (see, for example, the book *"Protective Groups in Organic Synthesis"* by Greene *et al.*).

In this formula, X_1 is an optionally-substituted heteroatom (i.e. an atom other than carbon). For example, X_1 may be oxygen, nitrogen or sulphur. If X_1 is nitrogen, the nitrogen is substituted with a third substituent, i.e. X_1 has the chemical formula NR_7. In a preferred embodiment, this third substituent (R_7) is hydrogen. Alternatively, R_7 (or any other optional substituent) may be OH, O-alkyl, O-heteroalkyl, alkyl or heteroalkyl.

In this formula, R_{24}, R_{25}, R_{26} and R_{17} are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl and a siloxane monolayer precursor-containing group, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl).

The isocyanurate functional group preferably has the following formula:

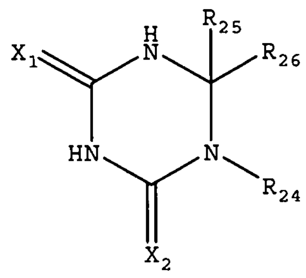


and derivatives thereof in which one of the NHs shown in the above formulae is either protected with a protecting group or is replaced by N-Halogen (preferably N-Cl or N-Br). Protecting groups are well-known in the art (see, for example, the book *"Protective Groups in Organic Synthesis"* by Greene *et al.*).

In this formula, X_1, X_2 and X_3 are independently-selected optionally-substituted heteroatoms (i.e. atoms other than carbon). For example, X_1 and / or X_2 may be oxygen, nitrogen or sulphur. If either X_1 or X_2 is nitrogen, the nitrogen is substituted with a third substituent, i.e. X_1, X_2 and / or X_3 have the chemical formula NR_7. In a preferred embodiment, this third substituent (R_7) is hydrogen. Alternatively, R_7 (or any other optional substituent) may be OH, O-alkyl, O-heteroalkyl, alkyl or heteroalkyl.

In this formula, R_{24} is selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl and a siloxane monolayer precursor-containing group, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl). R_{24} may be a protective group for NH.

The triazinedione functional group preferably has the following formula:



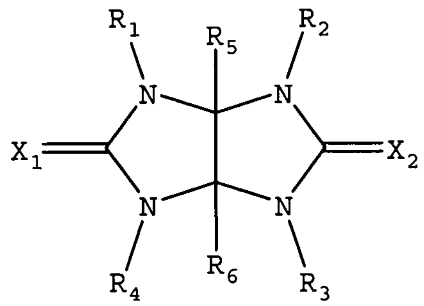
and derivatives thereof in which one of the NHs shown in the above formula is either protected with a protecting group or is replaced by N-Halogen (preferably N-Cl or N-Br). Protecting groups are well-known in the art (see, for example, the book *"Protective Groups in Organic Synthesis"* by Greene *et al.*).

In this formula, X_1 and X_2 are independently-selected optionally-substituted heteroatoms (i.e. atoms other than carbon). For example, X_1 and / or X_2 may be oxygen, nitrogen or sulphur. If either X_1 or X_2 is nitrogen, the nitrogen is substituted with a third substituent, i.e. X_1 and / or X_2 have the chemical formula NR_7. In a preferred embodiment, this third substituent (R_7) is hydrogen. Alternatively, R_7 (or any other optional substituent) may be OH, O-alkyl, O-heteroalkyl, alkyl or heteroalkyl.

In this formula, R_{24}, R_{25} and R_{26} are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl and a siloxane monolayer precursor-containing group, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl). R_{24} may be a protecting group for NH.

Preferably, the compound containing at least two hydrogen atoms is a glycoluril compound. In this case, the free NH is able to react with the siloxane precursor cross-linking compound. Alternatively, a group appending the glycoluril functional group may be capable of reacting with the siloxane precursor cross-linking compound. If one or more halogen atoms are not appended to any of the other atoms in the glycoluril functional group, then preferably the glycoluril functional group contains at least two NH groups (the second one is then activated to become NCl).

Preferably, the glycoluril compound of this second aspect has the following chemical formula:



In this formula, X_1 and X_2 are independently-selected optionally-substituted heteroatoms (i.e. atoms other than carbon). For example, X_1 and / or X_2 may be oxygen, nitrogen or sulphur. If either X_1 or X_2 is nitrogen, the nitrogen is substituted with a third substituent, i.e. X_1 and / or X_2 have the chemical formula NR_7. In a preferred embodiment, this third substituent (R_7) is hydrogen. Alternatively, R_7 (or any other optional substituent) may be OH, O-alkyl, O-heteroalkyl, alkyl or heteroalkyl.

In this formula, R_1, R_2, R_3 and R_4 are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl and heteroalkyl. R_5 and R_6 are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl and a siloxane monolayer precursor-containing group, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl). At least one of R_1, R_2, R_3 and R_4 is hydrogen. If none of R_1, R_2, R_3 and R_4 are a halogen (preferably chlorine or bromine) or a protecting group for NH, then preferably at least two of R_1, R_2, R_3 and R_4 are hydrogen. Protecting groups are well-known in the art (see, for example, the book *"Protective Groups in Organic Synthesis"* by Greene *et al.*).

One advantage of using this method is that it is simpler and more convenient than the first aspect. Simply, all that has to be done is for the precursors to be mixed together rather than the synthesis of a specially-designed molecule incorporating both a glycoluril group and a siloxane monolayer precursor group. As such, this also represents an improvement on the methodology in US 6969769, so this method can be used without the active application of microwaves to form a conventional coating on a substrate (i.e. a coating that is not effective against toxins but can be used for other purposes).

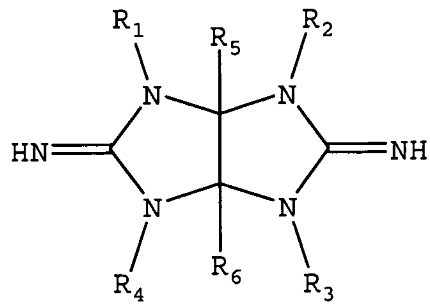
In addition, this method forms a resilient coating on the surface of a substrate. In particular, the functional compound is linked directly to the silicon centre rather than through a linker group and the possible *in situ* formation of the coating allows the functional group to be more resiliently attached to the surface. This resilient coating may be further enhanced by treating the substrate with microwaves (the specifics of which are the same as those described in the first aspect of the invention), such as substantially at the same time as applying the compounds to the surface.

Preferably, the functional compound is selected from a compound containing a functional group selected from the group consisting of glycoluril, isocyanurate and triazinedione. In particular, these functional groups contain three or more aliphatic nitrogens containing potentially free hydrogens, thus allowing each functional group to be attached to a surface through more than one silicon atom while exhibiting disease-control properties. Thus, a more resilient disease-control coating may be formed.

Other preferred features of this second aspect are the same as those of the first aspect. For example, the surface coating, once formed, is treated in the same manner as in the first aspect to make N-halogen groups (for example N-Cl or N-Br groups).

In a third aspect, the present invention provides a substrate having a coating deposited according to the first or second aspect. In particular, depositing a coating using microwaves rather than heat results in a physical change in the properties of the coating. Therefore, a coating formed while being irradiated by microwaves is physically different from a coating formed simply by heating. Accordingly, the third aspect provides a substrate having compounds as defined in either the first or second aspects attached to, and / or organised into an array on the surface of the substrate.

In a fourth aspect, the present invention provides a novel glycoluril derivative that is especially adapted for use in the first and second aspects of the present invention. This glycoluril derivative has the chemical structure:



(6)

wherein:

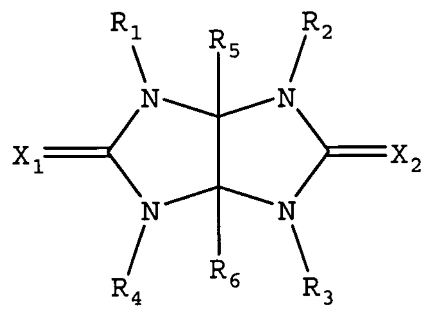
R_1, R_2, R_3 and R_4, R_5 and R_6 are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl, a group containing a siloxane monolayer precursor, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl), and

at least one of R_1, R_2, R_3, R_4, R_5 and R_6 is a group containing a siloxane monolayer precursor.

This compound has been found by the inventor to be particularly effective when attached to a surface and used to deactivate toxins. This compound may be formed from the corresponding glycoluril compound using techniques well known to the person skilled in the art for converting a C=O moiety into an N=H moiety (e.g. a ketone into an imide).

The preferred features of R_1 to R_6 are the same for this aspect as for the first and third aspects of the present invention.

In a fifth aspect, the inventor has found that the use of a siloxane monolayer precursor to attach a glycoluril functional group to a surface has certain disadvantages. Therefore, the inventor has sought new ways of forming disease-preventing coatings containing a glycoluril functional group on a surface. Accordingly, in a fifth aspect, the invention provides certain precursors for forming a coating containing a glycoluril functional group that is effective against toxins. These precursors have the following chemical structure:



(7)

R_1 to R_6 are as defined previously. At least one or R_1 to R_6 is a group containing a vinyl group, an imide, an acrylate, an alkene, an epoxide or an alkyl halide.

These precursors are more convenient to prepare than their siloxane monolayer precursor equivalents. These compounds may be prepared by the person skilled in the art using the techniques of the art.

In a sixth aspect, the present invention provides a method for forming a coating on a substrate by applying the compound (7) to a surface and allowing it to form a coating on the surface. The coating may be preferably formed or cured by heat or microwaves.

In a seventh aspect, the present invention provides a substrate coated with a coating formed by the sixth aspect of the present invention.

The present invention also provides the use of the coatings described above in the deactivation of toxins. These coatings may be formed from, for example, a compound containing a glycoluril functional group and a siloxane monolayer precursor group or compound (7).

Although preferred features described above have been described in relation to certain embodiments, it will be readily apparent to the person skilled in the art that preferred features of one embodiment are readily applicable to the other embodiments.

Although preferred features described above have been described in relation to certain embodiments, it will be readily apparent to the person skilled in the art that preferred features of one embodiment are readily applicable to the other embodiments.

EXAMPLES

The invention will now be further described by way of examples.

In one test process the synthesis of glycoluril crosslinked aluminium oxide may be achieved where Boehmite aluminium oxide is wetted with a basic water-alcohol solution containing glycoluril and tetraethylorthosilicate. The boehmite may then be irradiated with microwaves for three one minute intervals, washed thoroughly, and allowed to dry.

The product may be chlorinated via 30 minute wash in 0.5% aqueous sodium hypochlorite, washed thoroughly, and dried overnight at room temperature under vacuum. The presence of oxidative chlorine may be confirmed via colorimetric reaction with potassium iodide and starch indicator solution.

The ability of the chlorinated glycoluril crosslinked boehmite to deactivate toxins was confirmed via standard enzyme inhibition studies using three representative toxin simulants, nitrobenzene nitroreductase, lysozyme, and laccase. In each case the chlorinated glycoluril boehmite significantly (greater than 99.9% reduction in enzyme activity in all cases) inhibited the enzyme activity compared to controls. The controls used were untreated boehmite, treated unchlorinated glycoluril boehmite, and the representative enzyme solution without a solid matrix.

Separately, the synthesis of hydantoinylated aluminium oxide may be confirmed by wetting boehmite aluminium oxide with a basic water-alcohol solution containing 1-hydroxymethyl-5,5-dimethyl hydantoin and tetraethylorthosilicate. The boehmite may then be irradiated with microwaves for three one minute intervals, washed thoroughly, and allowed to dry. The product from this may be titrated via iodometry. Test results may be found to contain 8 ppm of active, oxidative chlorine. From previous experiments it is believed that 2 ppm or greater of active chlorine is suitable to inactivate toxins, with a greater amount of active chlorine resulting in a greater efficacy.

Unless stated to the contrary, procedures are carried out at room temperature (about 20°C) and atmospheric pressure (1 atmosphere).

CLAIMS:

1. A method for forming a coating for disease-control on a substrate, the method comprising:

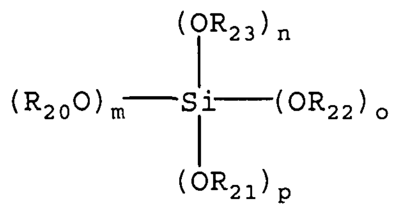
providing a compound containing at least two hydrogen atoms attached to one or more nitrogen atoms and a compound containing a cross-linking siloxane precursor group, and

applying the compounds to the surface.

2. The method of claim 1, further comprising exposing the surface to microwave irradiation.

3. The method of claim 2, wherein the coating formed is a coating for deactivation of toxins.

4. The method of any one of the previous claims, wherein the siloxane precursor cross-linking compound is a compound having the following structure:



where R_{20}, R_{21}, R_{22} and R_{23} are independently selected from hydrogen, an alkyl group or a heteroalkyl group.

5. The method of claim 4, wherein the siloxane precursor cross-linking compound is tetraethylorthosilicate ($Si(OCH_2CH_3)_4$).

6. The method of any one of the preceding claims, wherein the compound containing at least two hydrogen atoms attached to one or more nitrogen atoms is a compound containing a functional group selected from the group consisting of hydantoin, imidazolidinone, glycoluril, iscyanurate and triazinedione.

7. The method of any one of the preceding claims, wherein the surface coating is treated to produce N-halogen groups, for example N-Cl and / or N-Br groups.

8. The method of claim 7, wherein the method further comprises: exposing the surface to a toxin to cause the deactivation of the toxin.

9. A method for functionalising a surface of a substrate, the method comprising:

(a) providing a silicon-containing compound having two or more leaving groups and a functional compound comprising at least one nucleophilic group;

(b) contacting the silicon-containing compound and the functional compound with a surface of a substrate having nucleophilic sites thereon; and

(c) exposing the silicon-containing compound, the functional compound and the surface of the substrate to microwave radiation.

10. The method of claim 9, wherein the functional compound has two or more nucleophilic groups to allow the functional compound to adhere to the substrate via more than one silicon-containing compound.

11. The method of any one of the preceding claims, wherein the silicon-containing compound is selected from orthosilicic acid, a tetraalkoxysilane, preferably tetramethoxysilane or tetraethoxysilane, a tetraacyloxysilane, preferably tetraformyloxysilane or tetraacetoxysilane, tetraminosilane, or a tetra(alkylamino)silane.

12. The method of any one of claims 9 to 11, wherein the functional compound is 3(3-triethoxysilylpropyl)-5,5-dimethylhydantoin.

13. A substrate having a coating attached to and / or organised into an array on its surface by the method of any one of claims 1 to 12.

14. A process for deactivating toxins, the process comprising:

exposing a toxin to a coating comprising an N-halogen group.

15. The process of claim 14, wherein the N-halogen group is contained in a compound containing a functional group selected from the group consisting of hydantoin, imidazolidinone, glycoluril, iscyanurate and triazinedione.

16. A method for forming a coating for deactivation of toxins on a substrate, the method comprising:

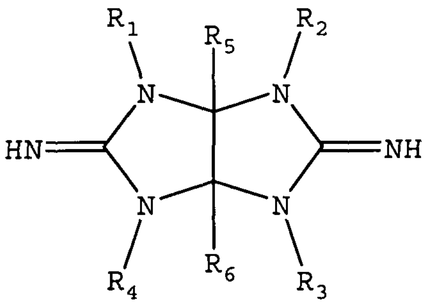
providing a compound containing a glycoluril functional group and a siloxane monolayer precursor group,

applying the compound to the surface, and

exposing the surface to microwave electromagnetic radiation.

17. A substrate having a compound containing a glycoluril functional group attached to, and / or organised into an array on a surface by being treated by the method of claim 16.

18. A compound having the structure:



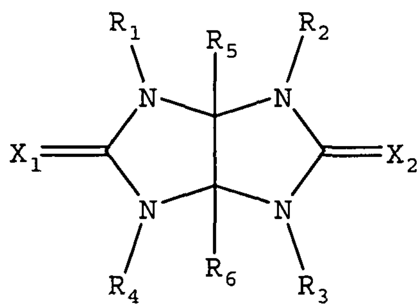
(6)

wherein:

R_1, R_2, R_3 and R_4, R_5 and R_6 are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl, a group containing a siloxane monolayer precursor, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl), and

at least one of R_1, R_2, R_3, R_4, R_5 and R_6 is a group containing a siloxane monolayer precursor.

19. A compound having the structure:



wherein:

X_1 and X_2 are independently-selected heteroatoms, optionally having one or more pendant independently-selected alkyl and / or independently-selected heteroalkyl groups and / or hydrogen,

R_1, R_2, R_3 and R_4, R_5 and R_6 are independently selected from hydrogen, the halogens (preferably chlorine or bromine), alkyl, heteroalkyl, OH, O-alkyl, O-heteroalkyl, NH_2, NH(alkyl), NH(heteroalkyl), N(alkyl)(alkyl), N(alkyl)(heteroalkyl) and N(heteroalkyl)(heteroalkyl), and

at least one of R_1, R_2, R_3, R_4, R_5 and R_6 is a group containing a vinyl group, an imide, an acrylate, an alkene, an epoxide or an alkyl halide.

20. A method for forming a coating for deactivation of toxins on a substrate, the method comprising:

providing the compound of claim 19,

applying the compound to the surface, and

exposing the surface to microwave electromagnetic radiation.

21. A substrate having a compound containing a glycoluril functional group attached to, and / or organised into an array on the surface by being treated by the method as defined in claim 20.

22. The use of a coating formed from a compound containing a glycoluril functional group and a siloxane monolayer precursor group in the deactivation of toxins.