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Water - emulsion composition based on triethoxysilylpropylamide perfluoro-2,5-dimethyl-3,6-dioxanonoic acid (Fluorosam-39) and perfluorocarboxylic acid as emulsifier

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Abstract: A new water -emulsion compositions were obtained based on organofluorosilicon compound «Fluorosam-39», giving high hydro- and oil-repellent properties to building materials. These compositions contain triethoxysilylpropylamide perfluoro-2,5-dimethyl-3,6-dioxanonoic acid, organic solvents - aliphatic hydrocarbons C₅-C₈, and as emulsifier - perfluoro-2,5,8-trimethyl-3,6,9-trioxadodecanoic acid. Limiting wetting angles of chalk stone by water and engine oil were 130 - 135° and 120 - 127° accordingly.

Keywords: water emulsion, triethoxysilylpropylamide perfluoro-2,5-dimethyl-3,6-dioxanonoic acid, organic solvents, limiting wetting angles.

Fluorosam-39 C₃F₇OCF(CF₃)CF₂OCF(CF₃)C(O)NH(CH₂)₃Si(OC₂H₅)₃, is widely applied for hydro- and oil-repellent proving of different building materials (chalk stone, bonding plaster, wood and others), also for works of art (monuments, historical buildings) against environmental hazards. Fluorosam-39 (F-39) compares favorably from organosilicon protecting compounds by more high oil-repellent properties, and from the compounds based on perfluorinated compounds, which one required the application of freons (for an example freon 113) as solvents. And these freons are prohibited by Montreal Convention. The synthesis and application of F-39 are described in many articles and patents [1-5]. Usually, it was used as 1-5% of solutions in organic solvents, most often in ethylacetate, however these solutions were not enough stable because of water presence in last one. The main disadvantage of the compounds based on F39 is their higher price in comparing with organosilicon compositions. There were developed emulsion composition in order to reduce the compound price based on F-39 and improve their ecological properties. These emulsion compositions are cheaper and more stable than solutions in ethylacetate. A range of solvents and emulsifiers were used for obtaining stable hydro- and oil-repellent compositions containing F-39. Aliphatic hydrocarbons C₅-C₈ of normal or iso-structure were tested as a perspective solvents that did not contain essential quantity of water like ethylacetate (Table 1). These

solvents are not miscible with water, they contain no more than 0.01% of mass moisture at 20°C. Solvents purity was «high grade» and were used without additional purification.

Table 1. Solvents for preparation of emulsions Fluorosam-39

Solvent	Boiling point, °C	Solubility parameters* δ (cal/cm ³) ^{0,5}
n-Pentane	36	7.05;7.0
Isopentane	28	7.05;6.7
n-Hexane	68.7	7.3
n-Heptane	98.4	7.45
n-Octane	135.7	7.55
Iso-octane	99.2	6.85
Petroleum ether	40-70	

*Askadsky A.A., Matveev U.I., «Chemical structure and physical properties of polymers», M., «Chemistry».1983, p. 231-232

It is turned that hydrocarbons C₅-C₇ and petroleum ether are very good solute F-39, generating stable solutions. Octane does not solute it.

Perfluorooxaalkylenecarbonyl acids based on Hexafluoropropylene oxide and its salts were tested as emulsifiers [6,7]. The application of salts did not give the formation of stable emulsions, whereas the acids were effective emulsifiers. As critical concentration of micelle formation (mole/l) of intermediates based on hexafluoropropylene oxide tetramer is lower in two times than based on trimer (2.29×10^{-4} and 1.15×10^{-2} mole/l accordingly), more effective acid based on Hexafluoropropylene oxide tetramer was used as emulsifier.

There were created a new effective hydro- and oil-repellent compound and water-emulsion compositions on its basis, which one are environmentally safe and stable upon storage [8]. This compound contained (mass.%): organofluorosilicon amide F-39 (20.0-69.8), aliphatic hydrocarbons C₅-C₇ with normal and/or iso-structure (29.9-79.80) and anion emulsifier (0.15-0.8). Perfluoro-2,5,8-trimethyl-3,6,9-trioxadodecanoic acid C₃F₇O[CF(CF₃)CF₂O]₃ - CF(CF₃) COOH was used as emulsifier. This compound could be store with no loss in quality and for a long time. A stable emulsion within several days was formed at its mixing with water in mass ration 1:(4-9).

Table 2. Hydro- and oil-repellent compositions, water-emulsion compositions on its basis, also limiting wetting angles date by water and oil M6-12g of chalk stone treated by these emulsions

Hydro- and oil-repellent composition mass. %			Solvent	Water-emulsion composition Content Water :	Limiting wetting angle, °	
F-39	solvent	emulsifier			By water	By oil
19.94*	79.76*	0.3*	n-hexane*	1:9*	132	108
29.9	69.8	0.3	n-hexane	1:9	136	119
29.9	69.8	0.3	n-hexane	1:4	137	121
69.8	29.9	0.3	n-hexane	1:9	114	106
30.0	69.85	0.15	n-hexane	1:9	134	129
29.76	69.44	0.8	n-hexane	1:9	137	121
69.8	29.9	0.3	n-heptane	1:9	136	127
69.8	29.9	0.3	Petroleum ether	1:9	131	125
69.8	29.9	0.3	Petroleum ether	1:4	131	127

*Limiting wetting angles by water are 117° and 109° accordingly at glass and aluminum treatment by this composition.

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