

FLUORINE CONTAINING POLYMER MATERIALS BASED ON UNSATURATED ACIDS MODIFIED ACCORDING TO ESTER FRAGMENT AND THEIR PRACTICAL APPLICATION

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Here we discuss the approach to the fluorine modification of acrylates and maleates ester fragment using partly fluorinated alcohols and acrylic, methacrylic and maleic acids' derivative synthesize them. We also discuss the reactions involving multiply bond and carboxylic group maleic acid fluorine containing mono-ester and acrylic acid esters. We analyze acrylic polymerization processes their co-polymerization with other olefines, and also fluorine containing polymers' characteristics. We also take a look at practical application fields of such polymer materials.

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4. Polymerization and Co-polymerization of Fluorine Containing Acrylates.

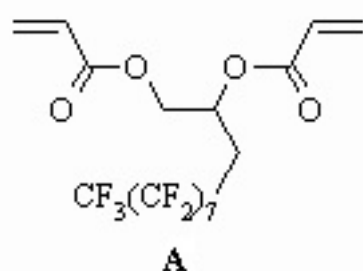
The polymerization of fluorine containing acrylates is going under radical initiators acting in water acetone solutions, as a rule in the majority of cases 2,2'- azo-bis-isobutyronitrile (AIBN). In the works [115-117] the polymerization of 2,2,3,3,3-pentafluoropropyl 4-fluoroacrylate and 2,2,3,3-tetrafluoropropyl 4-acrylate under the influence of 2,2'-azo-bis-(2-methylpropionamidine)dichloride at rather low temperature of 60-70 °C

described. Dodecylmercaptan is used as additive during the polymerization. In the work [119] the polymerization of perfluorooctylethylacrylate and methylacrylate in the presence of 2,2'-azobis(methylpropionamidine)dichloride (1-dodecanemercaptan, 160 °C, 1 min, after that at room temperature 1 hours) [118] or the one involving stearylacrylate in the presence of azo-bis-iso-butyramidine hydrochloride laurylmercaptan, 60°C, 3 h) [119] were studied. The polymerization of perfluorooctylethylmethacrylate passes the same way [120]. In these cases the yield of polymers is rather high and amounts to about 80%. Perfluorooctylethylmethacrylate and perfluorooctylethylacrylate polyacrylate is obtained out of corresponding monomers by 2,2-azobis-(2-amidinopropane)dihydrochloride (V-50) initiator action in the presence of bromooctadecyltrimethylammonium [121], and it is also obtained during the reaction of $\text{CH}_2=\text{CHOC}(\text{O})\text{CH}_2\text{CH}_2(\text{CF}_2)_n\text{CF}_3$ ($n = 5-11$) acrylate and aceto-oxy-methylmethacrylate by azobis(isobutylamidine dihydrochloride action in the presence of dodecylmercaptan and $\text{C}_{16}\text{H}_{33}\text{NMe}_3\text{Cl}$ at 60 °C [122].

Liquid-phase process carrying out is also used in the gamma radiation field. For example, in the work [123] the influence of fluorine atoms introduction into acrylates ester fragment is studied for the results of polymerization (the process was held at 313 K, the ^{60}Co γ -radiation). Kinetics of fluorine containing methacrylates polymerization is described quite well by kinetic scheme containing quadratic branching of progressing macromolecules and clearly defined gel-effect. Kinetics drastically differs from hydrocarbon methacrylates polymerization by absence of quasistationary conditions. Authors suppose, that at the beginning of fluorine containing acrylates polymerization the strong branching of structures takes place, leads to special isolation of growing macrocycles. The consequence of this is that there is no quasistationary stage of polymerization. Polymers based on fluorine containing acrylates are characterized by high elasticity and are closing raw rubber by their characteristics, while the polymers of fluorine containing methacrylates are fragile. It can be used to obtain polymer materials with new interesting characteristics.

Co-polymerization of fluorine containing acrylates and unsaturated compounds is one of the approaches to modified polymer materials synthesis. Mainly, the co-polymerization is held involving hydrocarboxylic acrylates and methacrylates and some of their derivatives in the presence of tetraalkylammonium bromide radical initiators (for example, octadecyltrimethylammonium bromide [121], laurylsulphate sodium in the aqueous ammonia medium [124], $\text{C}_{16}\text{H}_{33}\text{NMe}_3\text{Cl}$ [122], tetrabutylammonium bromide, dodecyltrichlorammonium chloride, $\{\text{C}_{22}\text{H}_{45}\text{N}[(\text{CH}_2\text{CH}_2\text{O})_{15}\text{H}]_2[\text{CH}_2\text{C}_6\text{H}_5]\}^+ \cdot \text{Cl}^-$ [119]).

Co-polymerization of hexafluoroisopropyl-trimethylacrylate and isobutyl vinyl ether goes effectively under the action of *tert*-butylperoxipivalate [125]. Co-polymerization of polyfluoroalkylacrylates $\text{CH}_2=\text{CHC}(\text{O})\text{OCH}_2\text{CH}_2(\text{CF}_2\text{CF}_2)_n\text{CF}_2\text{CHF}_2$ ($n = 3,4,5$) and butyl- or methylacrylate in the presence of AIBN [126-128] or system $\text{CuBr}/\text{CuBr}_2/1,10\text{-phenanthroline}/\text{ethyl-2-bromoisobutyrate}$ [129] goes effectively. $\text{CH}_2=\text{CHC}(\text{O})\text{OCH}_2\text{CH}_2(\text{CF}_2)_n\text{CF}_3$ ($n = 5-11$) acrylate co-polymerizes with methylmethacrylate, hydroxyethylmethacrylate [65,66,130] or styrene [131] in presence of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ and laurylmercaptane [132]. This co-polymerization goes under the action of AIBN in the presence of dodecanthiol [118,132] in acetone under the influence of AIBN [131], and co-polymerization of $\text{CH}_2=\text{CHOC}(\text{O})\text{CH}(\text{CH}_2\text{Cl})(\text{CF}_2)_n\text{F}$ ($n = 6,8$) acrylate and *N*-methylolacrylamide passes under the influence of $\text{K}_2\text{S}_2\text{O}_8 - \text{NaHSO}_3$ [90]. Co-polymerization of $\text{CF}_3\text{CF}_2(\text{CF}_2)_n\text{CH}_2\text{CH}_2\text{OC}(\text{O})\text{CH}=\text{CH}_2$ ($n = 2,4,6,8,10,12$) with methacrylate, polyethyleneoxide, dialkylaminoethylmethacrylate passes under the action of acetic acid peroxide at 60 °C [133], the polymerization of the first mentioned compound and stearylmethacrylate and 2-hydroxyethylmethacrylate passes at 65 °C [124], it also passes together with acrylates and silicon derivatives [134], and aminoganylpolsiloxanes [135], with cyanoethylcellulose [136]. *1H,1H,9H*-hexadecafluorononyl methacrylate and methacryloxypropyltrimethylsilane produce polymer, which is used to obtain coatings for textiles and [137]. The co-polymerization of $\text{CH}_2=\text{CHC}(\text{O})\text{O}(\text{CH}_2)_{11}(\text{CF}_2)_7\text{CF}_3$ and 2-hydroxyethylmethacrylate in presence of dibutylphthalate produces polymer product [138], and co-polymerization of $\text{CF}_3\text{CF}_2(\text{CF}_2\text{CF}_2)_n\text{CH}_2\text{CH}_2\text{OC}(\text{O})\text{CMe}=\text{CH}_2$ ($n = 6,7,8$) and $\text{CH}_2=\text{CHC}(\text{O})\text{O}(\text{CH}_2\text{CH}_2\text{O})_m\text{H}$ ($m = 3$) produces fluorine containing polymer [139,140], used for textile treatment. Polymerization of $\text{CH}_2=\text{CHOC}(\text{O})\text{CH}_2\text{CH}_2(\text{CH}_2\text{CH}_2)_n\text{R}$ ($\text{R} = 1\text{H},1\text{H}$ -dihydroperfluorooctyl, $n = 0-3$) is initiated by sodium metabisulphite and ammonium persulphate at 60 °C [141], and polymerization of *1H,1H,5H*-octafluoropentylacrylate in dimethylformamide initiated by hydrogen peroxide at 60 °C (6 h) [142]. Acrylates like $\text{CY}^1\text{Y}^2=\text{CXOC}(\text{O})\text{QR}_\text{F}$ ($\text{R}_\text{F} = \text{C}_1\text{polyfluoroalkyl}$; $\text{Q} = \text{bivalent organic group}$; $\text{X} = \text{H, F, CF}_3$; $\text{Y}^1, \text{Y}^2 = \text{H, F}$) are polymerized in $(\text{C}_5\text{Me}_5)_2\text{Sm}(\text{III})\text{Me}(\text{THF})$ [143], and *1H,1H,5H*-trihydrooctafluoropentylacrylate is polymerized in dimethylformamide medium at 80 °C (6 h) by hydrogen peroxide [142]. Co-polymerization of dimethylhydrosiloxane and **A** acrylate at 80 °C (6 h) in the presence of hydrosilylation catalyst results in formation of polymer product used to obtain glass coatings [144].



We should remember, that in most cases such polymer materials consist of many components, the interaction character of components between each other and polymer matrix is studied not well enough. That's unexpected effects can appear. For example, the quality of coating can increase because of synergism or on the contrary, it can decrease because of strong catalyzing action of one of mixture's components. Finding ways to increase adhesive characteristics of fluoroorganic polymer coatings is one of the most important practical goals of synthetic chemists. Here we should take into account economic factors. Thus, *1H,1H*-dihydroperfluoroalkylethylacrylates compare to *1H,1H,2H*-trihydroperfluoroacrylates are more expensive and less available, at that in a number of cases properties of their polymer are close to each other, that was used to achieve some practical goals. Lately co-polymers of these two groups of compounds are used bringing the mixture's content of more expensive component to 30-35 % [145].

The properties modification opportunities of fluorine containing polymer materials with their unique characteristics in case of wide application can lead to noticeable progress in many fields of techniques and medicine.

5. Practical Application Fields of Fluorine Containing Polymer Materials Based on Fluorine-Modified Acrylates and Maleate

One of the main practical application directions of fluorine containing polymer materials based on fluorine-modified ether part of acrylates and methacrylates molecules like $\text{CH}_2=\text{CRO}(\text{O})(\text{CH}_2)_n(\text{CF}_2)_m\text{A}$ ($\text{R} = \text{H, Me}$; $n = 1, 2$; $m = 1-10$; $\text{A} = \text{F, CF}(\text{CF}_3)_2$) and $\text{CH}_2=\text{CRO}(\text{O})\text{CH}_2(\text{CF}_2\text{CF}_2)_n\text{H}$ ($\text{R} = \text{H, Me}$; $n = 2, 4, 6, 8, 10, 12$) is creation of chemicals, which use for treatment of fiber surface makes them high hydro- and oleophobic, that increases water- and oil-repellent properties and creates protective surfaces based on different compounds. Thus, these compositions are used for treatment of cotton [118,121,146,147], silk [127], cellulose [139,148], synthetic fibers (nylon [122,149], viscose [141,150], polyester [151-153]) to add water- and oil-repellent properties to fabrics to provide them with complex of high consumer characteristics. The fabrics chemical treatment doesn't decrease the resistibility of fiber dyeing, the dirt gotten on to fiber can easily be deleted during washing, such treatment also forwards the improvement of textile articles stability of shape, especially wrinkle resistance. They are used for production of low humidity glass fiber [145].

Fluorine containing co-polymers in combination with other addition agents found their application in cosmetics production (skin moisturizers and tonics) [126,128,152-158] and they are also used for production of biomedical vessels and prosthesis including implantants [159]. High fluorinated organic compounds usually dissolve molecular oxygen. This particular feature increases quality of cosmetics.

Co-polymers based on *1H,1H*-dihydroperfluoroalkylacrylates and octadecylacrylate were used to create double coating of polyethylene film, that made it having fog characteristics [160]. Fluorosilicone polymers obtained using perfluoroalkylacrylates were used to modify surface and characteristics of polyvinylchloride widely used for agriculture [161]. Free surface energy of fluoro-silicone co-polymer amounted to 8-10 dyne/cm when molecular mass average value is 3000-7000, and this amount was 15-40 dyne/cm for double polyvinylchloride film depending on concentration of used co-polymer.

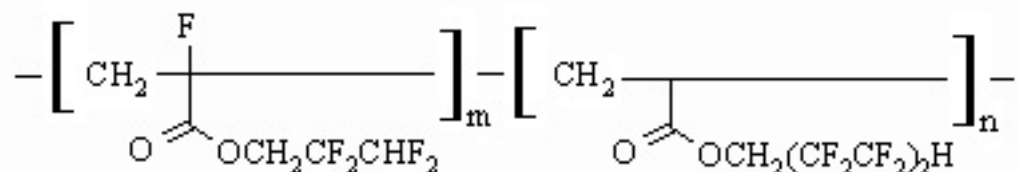
Co-polymers based on perfluorooctylacrylate and 3-(perfluorooctyl)propan-1,3-diol were used for membrane coating, which distinguished by high water-oil-repellency and firmness [162]. Co-polymer of *1H,1H,2H*-heptadecafluorodecylethyl acrylate and dimethylsiloxanediol applied on membrane, made of poly(methacrylate) and poly(dimethylsiloxane) created filtering base for separating water from benzene (benzene concentration within the range from 0.05 to 70 %) [163]. Here important feature introduced by perfluoroalkyl group is increase of polymer's gas impermeability. Co-polymerization of *1H,1H*-dihydroperfluorobutyl- and *1H,1H*-dihydroperfluoropropylperfluoromethoxyacrylates and unsaturated compounds, for example butadiene, produces products with significantly increased ability to swell in solvents [164], the lengthening of carbon chain conversely decreases this feature of polymer products.

Polymer based on fluorine containing acrylate $(\text{C}_n\text{F}_{2n+1})(\text{CH}_2)_x\text{CHROC}(\text{O})\text{CH}=\text{CH}_2$ ($n = 6, 8, 10$; $x = 0, 1$; $\text{R} = \text{H, CH}_2\text{OC}(\text{O})\text{CH}=\text{CH}_2$) produces film on the surface of membrane made of polytetrafluoroethylene, which possesses high water- and oil-repellent characteristics [165].

Poly(perfluorooctylethylacrylate) was applied in the composition to produce artificial marble with long life

water-repellent characteristics [166], and co-polymer of 1*H*,1*H*,2*H*,2*H*-heptadecafluorodecyl acrylate 1*H*,1*H*,5*H*-octafluoropentyl acrylate with dimethylaminoethylmethacrylate was applied in the composite mixture for glass coating, possessing antipolluting and antifog characteristics [167].

H(CF₂CF₂)_nCH₂O fragment in the polymer composition arouses changing of its few characteristics. The modified poly(methylacrylate) especially co-polymer of 1*H*,1*H*,3*H*-trihydrotetrafluoropropyl-2-fluoroacrylate with 1*H*,1*H*,5*H*-trihydroctafluoroamylacrylate possesses characteristics of polymer optical fiber [7].

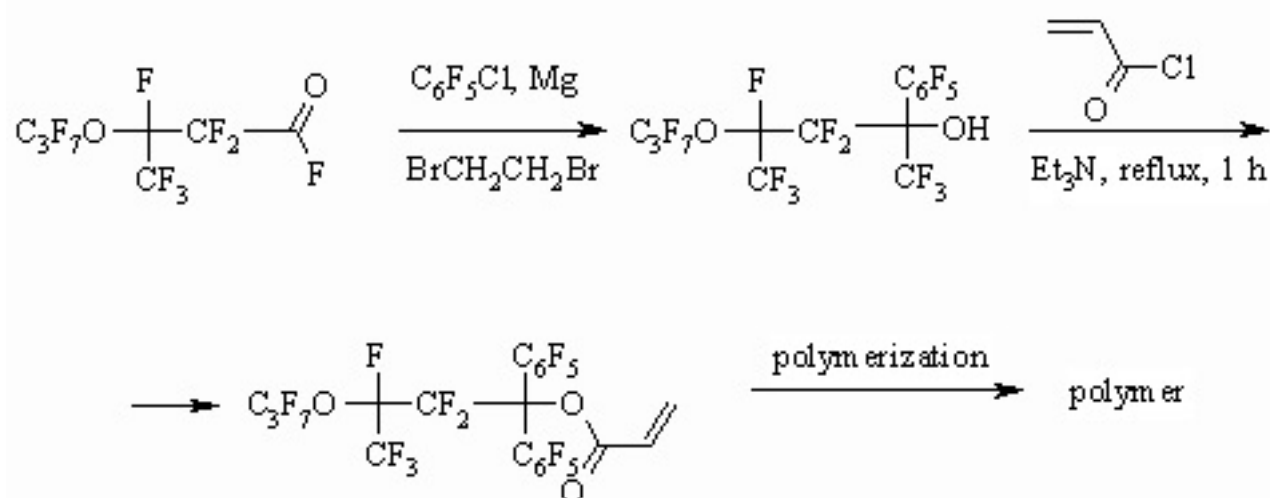


Refraction index of polymer optical fiber is rather low ($n_D^{20} = 1.4$), flow index of melt for applying a light reflecting covering is 10-30 g /10 min at 190 °C. They are well adhesion to the nucleus of this co-polymer, also their elasticity is high. This allows to obtain optical fiber of 2 mm diameter, standing strong flexure.

Polymers of α -fluoroacrylates exceed a lot in terms of many operational characteristics their methacrylate analogues. Introduction of fluorine into ester part of polymer resulted in improving of block organic properties characteristics and first of all it had an effect on thermal endurance and impact resistance (Table). Excellent optical properties of such fluorine modified polymers coupled with high thermal endurance, flexibility and wear resistance allows to use them in fiber optics. Refraction index of such polymers equal to 1.36 - 1.50 allows to use them as materials of optical fiber nucleus and light-reflecting covering [168-176]. They can be require materials for optical storage of optical disks [171,177].

Table. Some of Fluorine Containing Acrylates		Characteristics		
CH ₂ =CYCOOR		k_n , % optical transmission	n_D^{20}	a_n , KJ/m ² impact elasticity
Y	R			
F	CH ₂ CF ₂ CF ₂ H	91	1,398	95
	CH ₂ (CF ₂ CF ₂) ₂ H	90	1,379	77
	C(CF ₂) ₂ CF ₂ CF ₂ CF ₃	90	1,330	60
Me	CH ₂ CF ₂ CF ₂ H	91	1,422	71
	CH ₂ (CF ₂ CF ₂) ₂ H	91	1,400	50
	C(CF ₂) ₂ CF ₂ CF ₂ CF ₃	90	1,346	62

Polymers based on CH₂=CHC(O)OZ acrylates (where Z is fluorine containing alkyl or polyfluorobenzyl groups) possess optical and physical characteristics, that allows to use them in optical devices as optical fiber [79]. The synthesis of such polymers was carried out according to the following procedure.



Transparent fluorine containing co-polymers of acrylates were used to obtain materials for plastic optical fiber and optical light guides [178].

Fluorine containing acrylates are used to create optical membranes of low refracting index and reflection factor (no higher than 1.4 %) [179]. Co-polymers based on 1*H*,1*H*,5*H*-trihydroperfluoropentylacrylate 2(perfluorooctyl)-ethylacrylate and alkylacrylates are thermally resistant and stable to solvent influence produce transparent coatings useful for optical materials [134]. Such polymers were used to create LC optical elements [180] and photo-receivers and air photography devices [181].

Co-polymers of 1*H*,1*H*,3*H*-trihydrotetrafluoropropylmercaptane (6) and 1*H*,1*H*,5*H*-trihydrooctafluoroamylmercaptane (7) were used to obtain materials for plastic optical fiber and optical light guides [178].

Polymers based on fluorine containing ethers of maleic acid are used for compositions of thermal resin coatings, for impregnation of cotton, wool, nylon, dacron, and fabrics made of glass fiber as water-, mud- and grease-repellent films [107].

Acrylates fluorine modified by ester group are used to obtain filtering materials by treating of porous materials with fluorine containing polymers [10]. Such filters are high water- and oil-repellent. They are used for producing of different semi-products for medical, researching, consumer and industrial applications. Mono-perfluoroalkylesters of methacrylic acid were used.

Esters of fluorine alcohols and saturated acids can be used as lubricating materials, plasticizers, hydraulic liquids, and also to obtain polymers and co-polymers which are insoluble in common solvents, used to oil coatings [193, 194].

A transfer to science intensive production can't be carried out without renewal of equipment, enforcement of industrial safety standards, protection of equipment, functioning under conditions of corrosion active influence. Providing of corrosion proofness of main industrial funds must become a main scientific and technical goal and an important subject for the field of ecological and man-caused safety management. Fluorine materials of wide application field should play the decisive part to solve this problem, because they possess a number of unique properties.

Conclusion

As we see from the experimental data listed above the fluorine containing chemicals based on fluorine derivatives of acrylic, methacrylic and maleic acids found their wide application for providing materials with water- and oil-repellent properties, which effectiveness is essentially higher than the ones of siloxane compounds. The application of coatings made of these materials, which showed high consumer characteristics has started. As the achieving of fluorine chemicals high activity is becoming more and more important factor in the development of fine organic synthesis, it is obvious that new approaches listed in this review will find a wide application to carry out aimed synthesis of potential compounds of wide practical application field. This class of compounds is of deep interest of not only scientific, but also of application aspects. Realizing the significance of fluorine materials is a guarantee for success of this chemistry field.

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