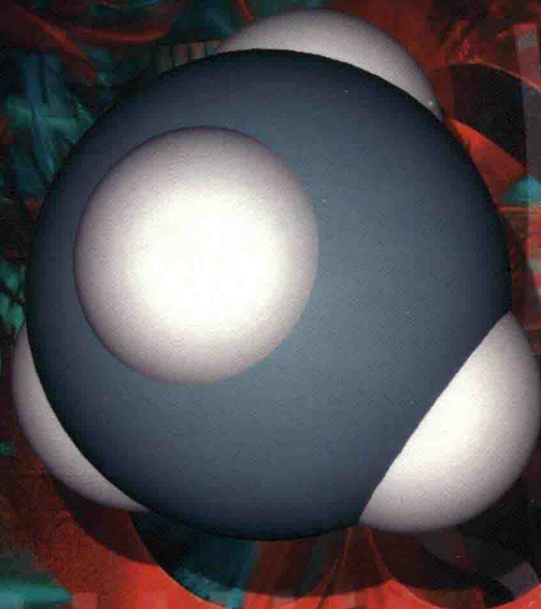
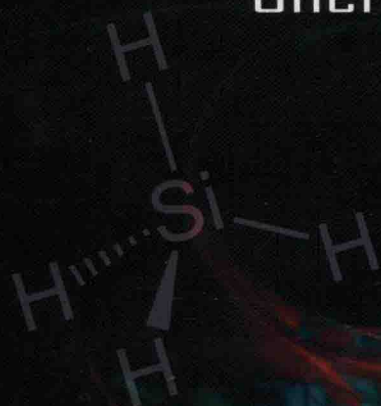


GASES - CHARACTERISTICS, TYPES AND PROPERTIES

SILANE

Chemistry, Applications
and Performance



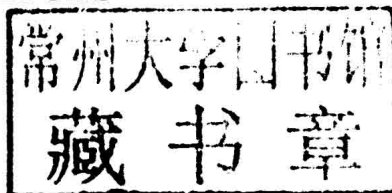
Katsuo Moriguchi
Susumu Utagawa
Editors

NOVA

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SILANE
CHEMISTRY, APPLICATIONS
AND PERFORMANCE

KATSUO MORIGUCHI
AND
SUSUMU UTAGAWA
EDITORS



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PREFACE

Silanes are synthetic compounds containing one or more Si-C bonds. They have wide application in industry, dentistry and medicine. They are used mainly as surface coating agents for surface modification and as coupling agents for adhesion promotion between two dissimilar materials. In this book, the authors present current research in the chemistry, applications and performance of silanes. Topics include the role silanes play in the processing of electronic chips and their thermo-chemical data; silane application in dental materials; thermal cationic polymerization of E-caprolactone using silanes; nanosilica surface modification by low-volatility silane coupling agents; and synthesis of a mesoporous silica hollow microsphere using methyltrimethoxy silane.

Chapter 1 - 2-Methacryloyloxyethyl phosphorylcholine (MPC) is a synthetic phosphorylcholine (PC) containing monomer which can polymerize or copolymerize with various vinyl monomers to synthesize MPC polymers having a wide variety of molecular architectures and functions. The MPC polymers have been widely used in surface modification of various substrate materials and devices to form cell-membrane-like surfaces, a kind of bioinspired biocompatible interfaces which have been shown to significantly resist nonspecific protein adsorption and cell adhesion. Among them, MPC copolymers incorporated with 3-methacryloxypropyl trimethoxysilane (MPTMSi) monomer unit, which is a typical methacrylate silane-coupling agent, have been specially designed for preparing biocompatible interfaces through a silanization process. In this chapter, we introduce cationic, neutral, and anionic MPC-MPTMSi copolymers and their applications for modification of glass-based substrates and devices, especially microfluidic devices which enable miniaturized and integrated chemical and biological applications.

Chapter 2 - In this chapter the status, trends, and challenges of working with silane and functional silanes are examined. The subject is far too vast to be covered in a single chapter, and much of what has been accomplished is proprietary and not available for review.

Thus the focus of this review is on standard practices and advancements published in the literature over the last twenty years; beginning with review of silane applications. Processes for formation and purification of silanes are reviewed with emphasis on the Direct, Siemens, Union Carbide/REC, Schmidt, and Ethyl/MEMC processes. The review continues with examination of the role silanes play in the processing of electronic chips, and analysis of hazards and public health issues one must be aware of when working with these compounds. The review concludes with examination of thermo-chemical data for silanes.

Chapter 3 - Silanes are synthetic compounds containing one or more Si-C bonds. They have wide application in industry, dentistry and medicine. They are used mainly as surface coating agents for surface modification and as coupling agents for adhesion promotion between two dissimilar materials. The role of silanes as either surface coating or coupling agents depends on their chemical structure. A wide variety of reactive organic functional groups can be bonded to the silicon atom such that silanes can react with the functional groups of many substrates. When silanes are used as a surface coating agent, the physical and chemical properties of the substrate surfaces are improved. As coupling agents, silanes enhance the chemical bonding between two dissimilar materials. Reaction mechanisms to illustrate these actions of silanes will be presented. Some techniques of surface treatment such as air-abrasion and chemical etching to enhance the adhesion between a silane and the substrate surfaces will also be covered. Findings from current studies of various silanes as surface-coating and coupling agents in different areas, with an emphasis on dentistry, will be discussed. The focus will be on the findings of performance of silanes as surface coating and coupling agents in different aspects of applications, including limitations in use. Finally, an outlook about on future developing trends of silanes in dental materials science will be provided. (219 words)

Chapter 4 - New initiating systems based on silanes and metal salts are proposed for an easy thermal cationic polymerization of ϵ -caprolactone at RT under air. They ensure good to excellent polymerizations. Moreover, a direct incorporation of the metal (Ag, Pd) through this one pot synthetic procedure is observed. The addition of small amounts of water or isopropanol allows a tuning of the polycaprolactone molecular weights. A coherent picture of the involved chemical mechanisms is presented.

Chapter 5 - Composites based on silicas and methyl methacrylate silane coupling agents are advanced materials for resin modification, thermoplastics, glass fiber reinforced epoxy resins, sensor technologies. The purpose of the present research is to combine silica-organic compounds with inorganic constituents to tailor the binding properties. It deals with a new method of covering silica nanoparticles by methyl methacrylate silanes in a pseudo-liquid state. The adsorption process and the chemical reactions of methacrylate-containing silanes and the surface of nanosized silica were examined by means of infrared spectroscopy, thermal analysis and transmission electron microscopy (TEM). The FTIR analysis confirmed the successful introduction of functional groups onto the nanosilica surface. The ratio of the parent free silanol and the grafted organosilyl groups was determined to be 1.4:1, i.e. 55% of the silane molecules react with the surface silanol groups in a monofunctional and 45% in a bifunctional way.

Chapter 6 - This chapter presents a new and facile method for the modification of alginate microcapsules using 3-aminopropyltriethoxysilane (APTES) in aqueous media. Spherical-shaped microcapsules (Alg-capsule) with carboxymethyl cellulose liquid core and calcium alginate shell were prepared by a conventional method, and then, Alg-capsules were modified to produce APTES/alginate hybrid microcapsule (AP-capsule) without using any organic solvents or precipitating agents. The optical observation showed that the texture of AP-capsules was more glossy and transparent than that of Alg-capsules. The surface morphology and elemental compositions of microcapsules were characterized by Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FT-IR) and X-ray Photoelectron Spectroscopy (XPS). The results suggest that APTES molecules were incorporated to the framework of the alginate shells via electrostatic interaction between

amino groups of APTES and carboxyl groups of alginate, and the hybrid shells had a thin and homogeneous structure. The shrinking of the capsule shells and the accumulation of APTES in the capsule shells were observed during the reaction between Alg-capsules and APTES molecules. This observation suggests the successful introduction of APTES molecules to the capsule shells as well as the self-condensation of loaded APTES molecules. In addition, the shell thickness and the amount of APTES in the capsule shells were found to depend on not only the pH of reaction solution but also the concentrations of APTES. From the permeability tests of compounds with different molecular weight (methyl viologen, nicotinamide adenine dinucleotide, diaphorase, bovine serum albumin), it was observed that the protein with 24 kDa easily permeated through Alg-capsule shells but hardly through AP-capsule shells. Therefore, AP-capsules were believed to have a molecular weight cutoff point of less than 24 kDa.

To demonstrate a practical application of AP-capsules, formate dehydrogenase (FDH) was selected as a model enzyme, and then the enzyme was immobilized with the capsules. The encapsulation of FDH in AP-capsules exhibited higher recycling ability than that in Alg-capsules due to the suppression of enzyme leakage. These results indicate that the AP-capsule is effective as a support of enzyme immobilization.

Chapter 7 - A mesoporous silica hollow micro sphere was synthesized in presence of methyltrimethoxy silane for the first time. The synthesized material was characterized by various instrumental techniques such as small-angle X-ray scattering, scanning electron microscopy, transmission electron microscopy.

Thermogravimetry/Differential thermal analysis, Fourier transform Infrared spectroscopy and Nitrogen adsorption measurement. The analysis reveals the material is mesoporous and having hollow micosphere morphology.

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Chapter 1

MODIFICATION OF GLASS SUBSTRATES AND MICROFLUIDIC DEVICES BY SILANIZATION OF BIOINSPIRED 2-METHACRYLOYLOXYETHYL PHOSPHORYLCHOLINE (MPC) POLYMERS FOR PREPARING BIOCOMPATIBLE INTERFACES

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ABSTRACT

2-Methacryloyloxyethyl phosphorylcholine (MPC) is a synthetic phosphorylcholine (PC) containing monomer which can polymerize or copolymerize with various vinyl monomers to synthesize MPC polymers having a wide variety of molecular architectures and functions. The MPC polymers have been widely used in surface modification of various substrate materials and devices to form cell-membrane-like surfaces, a kind of bioinspired biocompatible interfaces which have been shown to significantly resist nonspecific protein adsorption and cell adhesion. Among them, MPC copolymers incorporated with 3-methacryloxypropyl trimethoxysilane (MPTMSi) monomer unit, which is a typical methacrylate silane-coupling agent, have been specially designed for preparing biocompatible interfaces through a silanization process. In this chapter, we introduce cationic, neutral, and anionic MPC-MPTMSi copolymers and their applications for modification of glass-based substrates and devices, especially microfluidic devices which enable miniaturized and integrated chemical and biological applications.

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1. SURFACE MODIFICATION OF SUBSTRATES OF MICROFLUIDIC DEVICES

1.1. Microfluidic Devices

Microfluidic devices, or the so-called lab-on-a-chip devices and micro-total analysis systems (μ -TAS), are systems that process or manipulate small amounts of fluids using channels with dimensions of typically sub-millimeter scale for miniaturized and integrated chemical and biological applications [1]. They offer the possibility for performing laboratory operations on a small (10^{-4} to 10^{-9} m) scale with considerably low (10^{-9} to 10^{-18} L) reagent consumption, fast response times, and better process control in comparison with their conventional laboratory counterparts [2-8]. They also offer the possibility of massive parallelization and integration of an entire laboratory on a single chip, thereby allowing samples to be analyzed at the point of need and diseases to be diagnosed at the point of care rather than at the conventional laboratories and hospitals [8-13]. Till now, microfluidic devices have become a distinct field with many appealing applications that could herald a new era of high integration and miniaturization in the fields of physics, chemistry, biology, medicine, pharmacy, and environmental science [1, 14-25].

1.2. Motivations of Surface Modification in Microfluidic Devices

The control of surface properties becomes an indispensable prerequisite for various applications of microfluidic devices. As the size decreases, the surface-area-to-volume ratio of a microfluidic device increases considerably. This certainly provides many unique merits in thermodynamics, reaction kinetics, and transport phenomena [1, 26, 27] but simultaneously magnifies the effect of nonspecific bindings, resulting in fluid anomalies and low performance [28-31]. This effect is especially unfavorable in microfluidic devices used in a broad range of biomedical applications ranging from immunoassays, DNA analysis, and cell diagnostics to highly integrated and miniaturized microfluidic systems, because nonspecific bioadsorption of proteins, DNAs, and cells degrades the assay sensitivity seriously.

Accordingly, the motivations for surface modification in microfluidic devices for successful biomedical applications can be classified into two groups: (i) reduce or control unwanted analyte-wall interactions, especially nonspecific protein adsorption and subsequent bioreactions; and (ii) construct versatile surfaces on a chip to realize particular functions, especially for example, control of electroosmotic flow (EOF) which is a state-of-the-art approach most widely applied in microfluidic devices for fluid delivering. To address these issues, different surface strategies using polymers [29, 32-42] have been adopted to modify the microchannels fabricated on substrate materials, including SiO_2 -based materials [14, 15, 26, 43] such as glass and quartz, and synthetic polymers [44-48] such as poly(dimethylsiloxane) (PDMS), poly(methyl methacrylate) (PMMA), and poly(ethylene terephthalate) (PET).

1.3. Strategies and Materials for Surface Modification in Microfluidic Devices

In general, polymeric substrates are slightly hydrophobic, leading to difficulty in liquid introduction and notorious nonspecific analyte-wall adsorption. Therefore, surface modifications of polymer-based microfluidic devices are mainly driven by efforts to increase the hydrophilicity of the surface. Various covalent and non-covalent polymer modifications have been used for polymeric microfluidic devices through surface chemistry and physics [32, 35, 36, 38, 40-42]. Surface grafting of different hydrophilic monomers on PMMA substrates for microfluidic applications has been reported by Stachowiak *et al.* [49]. Results indicated that photo-grafted layers of PEG methacrylate (PEGMA) demonstrated the ability to substantially reduce nonspecific protein adsorption. Surface activation is commonly employed to oxidize and render hydrophilicity to PDMS surfaces. The reactive silanol functional groups generated by surface activation (for example, oxygen plasma treatments) can be used for subsequent surface chemistry such as silanization and various polymerizations. Hu *et al.* have grafted acrylic acid, acrylamide, 2-hydroxyethyl acrylate, dimethylacrylamide, and PEG-monomethoxyl acrylate onto PDMS to yield hydrophilic surfaces [50]. The grafted surfaces exhibited greatly reduced adsorption of proteins and retained EOF mobility and stability upon exposure to air as compared to oxidized PDMS. Recently, Lin *et al.* have reported grafting of epoxy-modified hydrophilic polymers onto a PDMS microfluidic chip to resist nonspecific protein adsorption and suppress EOF [35].

For glass microfluidic devices, procedures for silanization of microchannels employing silane reagents in combination with different hydrophilic polymers have been commonly employed to suppress nonspecific protein adsorption and control EOF. Channel walls are covalently coated with hydrophilic polymers such as polyacrylamide, poly(ethylene glycol) (PEG), and poly(vinyl alcohol) (PVA) via a silane sublayer, which is first deposited on the wall by derivatization using silane reagents [29, 33, 34, 37, 39]. Surfaces with permanent, robust, and reproducible coverage have been achieved with these covalent polymer coatings, and simply-formed and regenerable modifications have been realized using polymers that get physically adsorbed to the microchannel wall. Physically adsorbed polymeric coatings can be prepared using neutral polymers such as PEG, PVA, and poly(N-hydroxyethylacrylamide) (PHEA) and their derivatives or using charged polymers such as polyethyleneimine (PEI) and chitosan. The use of charged polymeric coatings makes it possible to both control EOF and improve protein separation in chip-based electrophoresis.

Recently, in addition to the aforementioned conventional polymer surfaces, a polymer family containing 2-methacryloyloxyethyl phosphorylcholine (i.e., MPC polymers) have also attracted considerable attention for modifications of both glass and polymer-based microfluidic devices because of their high performance in the suppression of nonspecific adsorption of various proteins, platelets, and cells (Figure 1). MPC polymers have been used to form cell-membrane-like interfaces for chip applications *via* physical adsorption [51-58], chemical bonding [59-65], and photo-induced grafting [66, 67]. In this chapter, we focus on the development and applications of MPC polymer biocompatible interfaces for modification of glass substrates and microfluidic devices through silanization processes.

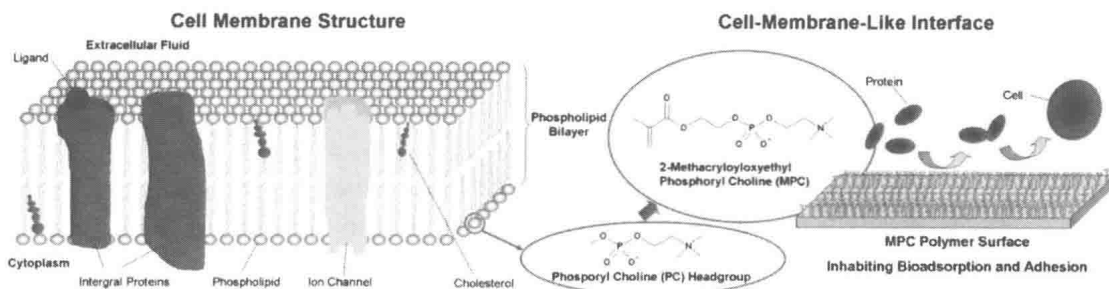


Figure 1. Schematics of cell membrane structure and cell-membrane-like surface with MPC polymer for inhibiting bioadsorption. (Image reproduced, with permission (Springer), from ref. 148).

2. BIOINSPIRED 2-METHACRYLOYLOXYETHYL PHOSPHORYLCHOLINE (MPC) POLYMERS

The cell membrane is the biological and nanostructured membrane separating the intracellular components from the extracellular environment. It consists mainly of amphipathic phospholipids, spontaneously assembling as a continuous, spherical lipid bilayer structure where the hydrophilic head (polar) regions shield the hydrophobic tail regions from the surrounding polar fluid of the cytosolic and extracellular environments, and are consequently located at the outer faces of the resulting bilayer.[68] Since it serves diverse biological and physicochemical functions in cells, the cell membrane structure has inspired novel ideas in the fabrication of biomaterials and biointerfaces having cell-membrane-like structures (Figure 1). One of the major phospholipid polar groups on the cell membrane is phosphorylcholine (PC), which has a zwitterionic head group. The monomers comprising the PC group have been synthesized with attention to the chemical structure of the phospholipid molecules [69-71] and their polymers (referred to as phospholipid polymers) have been applied in the preparation of cell-membrane-like structures [72-78]. In 1990, Ishihara *et al.* reported the significant functions of phospholipid polymer materials in the biomedical field and successfully synthesized phospholipid monomers with high yield and excellent purity [79]. The representative monomer, MPC, is a methacrylate with a PC unit as a phospholipid polar group (Figure 1). Currently, the MPC monomer is industrially produced and distributed worldwide by a Japanese company. Many phospholipid polymers based on MPC polymer chemistry have been developed and studied for functionalized surface modification. The MPC monomer can copolymerize with various vinyl monomers to form phospholipid polymers having a wide variety of molecular architectures including random [53, 79, 80], block [81-83], graft [66, 67, 84], charged [59-61, 85], and end functional polymers [58, 62, 86-88]. They could form cell-membrane-like surfaces by coating the polymer [59-61, 89, 90], blending with the polymer [87, 88, 91, 92], and grafting to the polymer chains [90, 93, 94], and thereby provided biointerfaces capable of suppressing many biological responses such as nonspecific interactions with proteins, platelets, and cells (Figure 1). In addition, with incorporation of functional units enabling bioconjugation, these MPC polymers could also form PC-covered surfaces capable of selectively interacting with specific biomolecules [51, 52, 88, 95, 96]. Accordingly, the researches and applications cover biomembranes [97, 98], artificial organs [99, 100], vehicles for drug delivery systems (DDS) [88, 96, 101-104], scaffolds for tissue

engineering [86, 88, 102, 105, 106], nanoparticles for immunoassays [95, 104], high sensitivity sensors [107, 108], and most recently microfluidic devices [51, 53, 54, 56, 59].

3. MPC POLYMERS DESIGNED FOR MODIFICATION OF GLASS-BASED SUBSTRATES AND MICROFLUIDIC DEVICES THROUGH SILANIZATION

3.1. Polymer Design

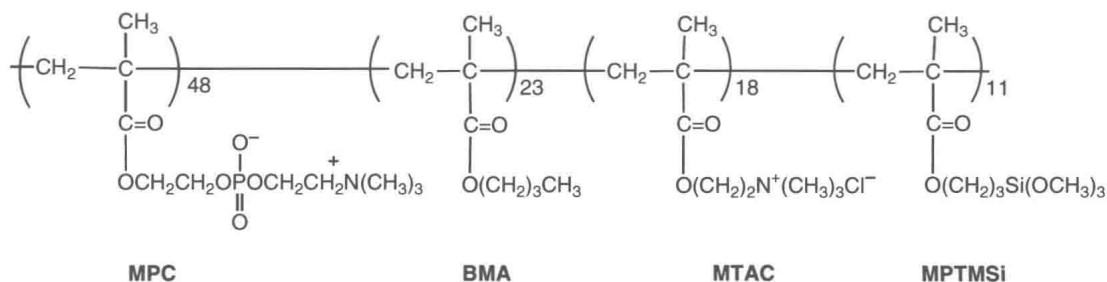
As a SiO₂-based material, glass is an initial and popular substrate to fabricate and develop microfluidic devices, especially for biological separation and analysis. This is not only because glass is chemically similar to fused silica and it is easy to transfer the classical separation behaviors from the conventional silica-based capillary electrophoresis (CE) to chip-based devices [109, 110], but also because their excellent properties such as physical rigidity, chemical inertness, optical transparency, thermal stability, and electrical insulation are very attractive and can satisfy some special chemical and analytical requisites, such as chemical syntheses, fluorescence detections, electrochemical analyses [43, 111]. As further downscaling of feature size to the nano scale, glass becomes an ideal substrate material to fabricate nanofluidic devices. This is because, in comparison with other popular substrate materials such as PDMS and other polymer-based materials, technologies for the fabrication of glass structures have already been well established in nanoscales with high resolution, reproducibility, and flexibility. Furthermore, the surface of glass is negatively charged and can generate a significant electroosmotic flow (EOF), which is favorable for some electrokinetic microfluidic devices [15, 112, 113].

The EOF is widely applied in miniaturized systems for fluid delivery [114, 115]. In some microfluidic systems with biological applications, proteins (or peptides) and cells are transported by the EOF. However, the irreversible, uncontrolled nonspecific protein adsorption and cell adhesion lead to low analytical efficiency and difficulty of EOF control, especially in the silica-based substrates, which make it particularly difficult to realize μ -TAS on glass for protein- and cell-related applications [33, 34, 37]. At the molecular level, rapid adsorption of proteins is generally thought as the initial stage of cell adhesion to a surface [77, 116, 117]. Therefore, prevention of protein adsorption is an indispensable prerequisite for successful biological applications in both protein- and cell-related microfluidic devices.

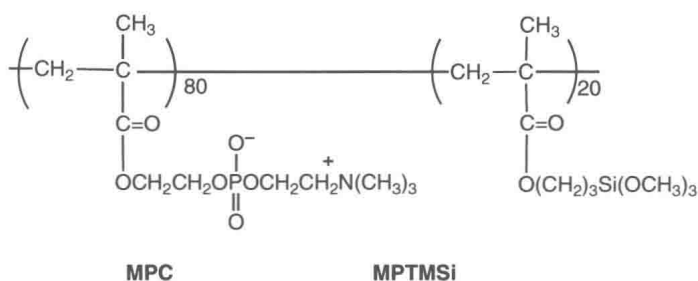
For surface modification of microchannels in microfluidic devices with polymers, several strategies such as filling coating [51, 53, 55, 59, 61], photo-induced polymerization [66, 67, 118], and electrospray deposition (ESD) [57, 58, 119, 120] have been developed. As an effective approach for suppression of protein adsorption, the strategy of surface coating has been widely applied. Till now, the coating materials for glass substrates and microchannels are mainly neutral, hydrophilic polymers, either covalently bound (such as polyamide and its derivatives), or physically adsorbed (such as dextran, poly (vinyl alcohol) and polyethylene oxide) [33, 34, 37]. They are effective for reduction of protein adsorption, but the EOF is usually dramatically reduced or totally eliminated simultaneously. For electrokinetically actuated microfluidic applications, it is unfavorable. In order to take advantage of EOF, charged polymer coatings are generally applied [121, 122]. However, the researches often showed that it is problematic, especially when charged proteins are handled on the surfaces

with opposite charges, where the potential of adsorption may arise because of electrostatic attraction [123-125]. Apparently, it is quite difficult to construct a charged surface having ability of suppressing both anionic and cationic protein adsorptions.

(a) PMBASi



(b) PMSi



(c) PMBSSi

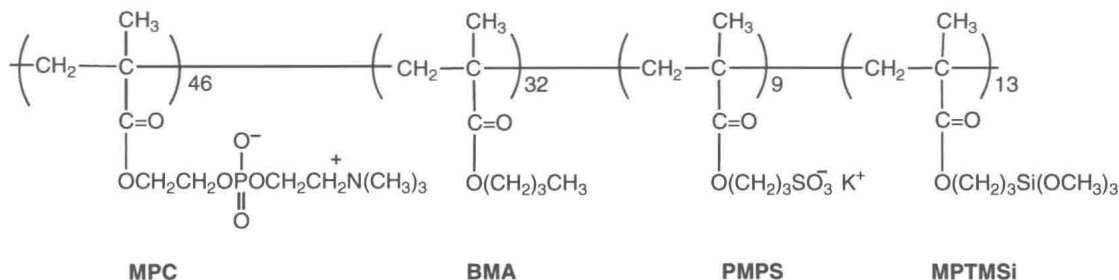


Figure 2. Chemical Structures of representative MPC polymers containing silane moieties and different charged moieties for glass microfluidic devices. (a) PMBASi (cationic), (b) PMSi (neutral) and (c) PMBSSi (anionic).

As introduced in the former section, the MPC polymer can be used to modify various surfaces, and their researches and applications cover wide areas from a bulk class to micro- and nano-scales accordingly. MPC polymers have been used in modification of substrates and microfluidic devices. For electrokinetic microfluidic devices on glass substrates, three representative MPC polymers containing silane moieties and different charged moieties were designed (Figure 2). They were poly(MPC-*co*-*n*-butyl methacrylate (BMA)-*co*-2-methacryloyloxyethyl trimethylammonium chloride (MTAC)-*co*-3-methacryloyloxypropyl trimethoxysilane (MPTMSi) (referred to as **PMBASi**, Figure 2a), poly(MPC-*co*-MPTMSi)