Effects of plasma treatment time on modification of acrylic denture material

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Abstract

Objective: To study the relationship between plasma treatment time and efficacy. Methods: Test specimens were prepared from an acrylic resin denture material in the size of 2 mm × 10 mm × 10 mm. Plasma treatment was carried out on the surface of Polymethyl methacrylate (PMMA) at different time. XPS studies, IR spectra studies and measurement of wetting angle were performed. Results: XPS showed the peak corresponding to C-O getting higher as the treatment proceeded, however at 120 seconds, the peak did not increase any longer and partly crossed with the peak at the duration of 60 seconds. IR spectra showed the wave corresponding to C-H was reduced as O2-plasma treatment proceeded, and then changed little. Wetting angle initially decreased dramatically, however, as the reaction proceeded, wetting angle increased slightly. Conclusion: Equilibrium was reached for introducing oxygen-containing groups and changing of C-H. As the treatment proceeded, wetting angle increased slightly.

Keywords: plasma; modification; acrylic denture material

INTRODUCTION

Because of the friable nature of the supporting mucosa, force concentration can result in tissue trauma and sore spots. As a result of these conditions, patients frequently do not wear dentures because of the discomfort that accompanies their use. To alleviate the possibility of discomfort arising from denture base force transfer to oral mucosa, many studies have been done [1-2]. Denture soft lining materials have been applied to the tissue surface of dentures to reduce local pressures and improve retention of the prosthesis [3-5]. One of the most serious problems with soft lining materials is the failure of adhesion between the soft lining materials and the denture base [6].

For the reasons of low surface energy, chemical inertness, surface contamination and weak boundary layer, polymer surface often presents low wettability and dissatisfied adhesion characteristics. Therefore, the surface of polymer needs to be modified.

The following methods are commonly used for polymer surface modification: chemical modification; actinnochemistry modification; surface modification by modifying agent; dynamochemical treatment; heat treatment; coupling agent modification and chemical modification by radiation.

Some of above-mentioned methods are too complicated to operate, and some produce a great deal of waste liquid during the process, which is harmful to health and environment. Low temperature plasma is a new subject based on the development of physics, chemistry, electronics and the vacuum technique. Low temperature plasma has characteristics of low reaction temperature, being easy to handle, low cost and without pollution to the environment [7-8]. Plasma treatment has previously been used in surface modification of medical devices and materials, by improving biocompatibility.

☆ This work was supported by Science Fund of Department of Education of Jiangsu Province(06KJD320125)

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and adhesion characteristics\cite{9-11}.

**MATERIALS AND METHODS**

The specimens were prepared with acrylic resin denture material (Zi Ran, Nissin, China) in the size of 2 mm × 10 mm × 10 mm. The surfaces of all the specimens were smoothed with aluminum oxide paper (Suzhou sand-paper factory) in 280 and 800 mesh, then washed with distilled water and dried in air. The specimens were placed in a glass tubular reactor. The reactor was evacuated from the right side using rotary vacuum pump until a pressure of 0.01 mmHg achieved. Oxygen gas was then introduced into the reactor to displace the residual gases. Evacuation and introduction were repeated several times. Finally, the pressure was controlled at 0.3 mmHg by appropriate opening of the inlet valve. Imposition of power of 60 w created the condition under which plasma treatment was carried out.

1. X-ray photoelectron spectroscopy studies: The power of 60 w was imposed for O\(_2\)-plasma treatment with different treatment time (10s; 30s; 60s; 120s), 2 specimens in each group. X-ray photoelectron spectroscopy studies were performed on an electron spectrometer (ESCALAB MK- II VG Corp, U.K.), with MgK\(\alpha\) X-rays (12.5 kv) having a background pressure lower than 1 × 10\(^{-7}\) Torr.

2. IR spectra studies: The power of 60 w was imposed for O\(_2\)-plasma treatment with different treatment time (0.5 min; 1 min; 2 min; 4 min; 8 min), 2 specimens in each group. IR spectra studies were performed on an IR spectrometer (NEXUS870, NICOLET Corp, U.S.).

3. Change of wetting angles: 42 specimens were prepared from an acrylic resin denture material in the size of 2 mm × 10 mm × 10 mm and randomly divided into 7 groups (6 specimens in each group). O\(_2\)-plasma treatment time was from 0 to 6 min. Wetting angles were measured with goniometer (JJC-1, Changchun experiment instrument Corp, China). The measurements were made with doubly distilled water at 5 points by the static sessile drop method.

**RESULTS**

*Fig 1* is C\(_{1s}\) XPS of control and O\(_2\)-plasma treatment groups on the surface of PMMA. The peak corresponds to structure of C-C at 284.5 eV and the peak corresponds to structure of C-O at 287 eV. At 287 eV, the peak got higher as the treatment proceeded, however at 120 seconds, the peak did not increase any longer and crossed with the peak at the duration of 60 s. *Fig 2* was amplified part of C\(_{1s}\) XPS. *Fig 3* is IR spectra of control and O\(_2\)-plasma treatment groups on the surface of PMMA. At 2960-2870 cm\(^{-1}\), the wave corresponding to C-H was reduced as O\(_2\)-plasma treatment proceeded, and then changed little. *Fig 4* showed the relationship between wetting angle and treatment time. Wetting angle initially decreased dramatically, however, as the reaction proceeded, wetting angles increased slightly.
DISCUSSION

The energy of particles in plasma is higher than the energy of ordinary bond (C-H; C-N; C-Cl; C-F; C=O; C=C; C≡C). This means plasma is powerful enough to break or realign the various chemical bonds, which make macromolecule degraded. The low temperature plasma can stimulate molecules and atoms effectively without damage of the molecules of the polymer[12,13].

Wettability is usually referred to the wetting process among liquid/air/solid. The wetting angle provides a quantitative measure of the wetting process[14,15]. A liquid is considered to be wetting a surface when $\theta$ is < 90 degrees, and is considered nonwetting when $\theta$ is > 90 degrees. A wetting angle $\theta = 0$ degree corresponds to perfect wetting, in which the drop spreads to from a film on the surface[16]. The smaller the wetting angle, the greater the wettability[17]. The ability of a solid to become wet is proportional to its surface energy. If adhesion between solid and liquid is stronger than cohesion within liquid, the solid can be wetted. On the contrary, the solid cannot be wetted. The wettability characters on the surface of polymer can be summed up as: The wettability of a polar compound is better than that of a compound without polar groups; Wettability of a polymer has relation with its composition. Adding other atoms into the chain of carbon can improve wettability of polymer remarkably, except the F atom. The order of atoms improving wettability is H<Cl<Br <I<O<N; Wettability of polymer is decided by characters on the surface of polymer can be summed up as: The wettability of a polar compound is better than that of a compound without polar groups; Wettability of a polymer has relation with its composition. Adding other atoms into the chain of carbon can improve wettability of polymer remarkably, except the F atom.

Plasma treatment can change chemical composition, increase surface energy, eliminate contamination and a weak boundary layer on the surface of the polymer, so as to improve its wettability and adhesion[18,19]. In this study, the peak corresponding to C-O at 287eV became higher as treatment proceeded, indicating a large number of oxygen-containing groups were introduced onto polymer surface[20,21]. At the duration of 120 seconds, the peak did not increase any longer and partly crossed with the peak at 60 seconds. The prolonged period of exposure leaded to excessive surface degeneration, which spoiled the surface properties due to accumulating of degenerated remnants[22]. This explained the reason for the initial decrease in wetting angle and then slight increase. IR spectra on O$_2$-plasma treated surface showed a similar tendency. At 2960-2870cm$^{-1}$, the wave corresponding to C-H was reduced as O$_2$-plasma treatment proceeded, and then changed little, indicating decrease in structure of C-H and increase in structure of C-C, which meant the cross-linking structure was formed on the surface of PMMA. Cross-linking structure is a kind of dense layer and effective in eliminating weak boundary layer on the surface of PMMA.

References


