Preparation and Superficial Characteristics of Light-Cured Resin Containing Negative Ion Powder

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Abstract. The objective of this paper is to prepare light-cured resin material containing negative ion powder, test and evaluate the performance of its composite material, and provide some experimental basis for its clinical application. Adopt mechanical agitation dispersion and ultrasonic dispersion for ultra-fine processing of negative ion powder. Prepare the antibacterial composite resin by evenly dispersing negative ion powder in 4% ratio of 0% to 2% in Durafill composite resin. Observe the dispersion uniformity of antimicrobial agents by scanning electron microscope (SEM), and use Fourier transform infrared spectroscopy (FTIR) to determine the curing rate of the modified composite resin after adding different proportion of negative ion powder. Test the microhardness of modified composite resin by Vickers microhardness meter. The SEM result shows that the negative ion powder was dispersed uniformly in the composite resin. The results of initial curing rate of composite resin showed that the addition of negative ion powder did not lead to the decrease of curing rate but increased the curing rate to some extent. The mechanical properties test showed that the surface hardness was decreased compared with the control group, and it was gradually decreased with the increase of the proportion of negative ion powder.

1. Introduction
The development of stomatology benefits from the progress of materials science. Compound resin has been widely used and developed since its application in dental prosthesis. In view of various problems in clinical application, the research on compound resin has been paid increasing attention[1]. Compound resin, especially light-cured compound resin, has excellent cosmetic effects[2]. With the development of material science, its performance has been greatly improved, the physical properties, such as wear resistance, have been close to enamel level two, and the clinician's operation next to the chair has become more convenient. As a result, compound resin has gradually become a widely used dental restoration material. However, microleakage and secondary caries are often formed between the compound resin material and dental tissue, which is the main factor leading to the failure of compound resin repair[3-4].

Negative ion powder is a cyclic silicate mineral consisting of SiO₂, FeO, Fe₂O₃, B₂O₃, Al₂O₃, Na₂O, MgO, Li₂O and MnO, which also contains trace elements such as Cr, Zr, Zn and Ti, which are beneficial to human body. Known as vitamins in the air, it regulates the balance between cation and anion, and inhibits the aging of human cells. Negative ions can also purify the air and deodorize. Some people also propose to add negative ion powder into toothpaste to make it produce a large number of negative ions in the process of brushing teeth, which has a certain positive effect on oral health care. The use of anion-producing materials has been developed from simple air and water treatment to various aspects of life. The manufacturing process has been processed by simple mechanical crushing to ultrafine and
nanocrystalline chemical treatment. High concentration of negative ions can effectively alleviate seasonal stress\textsuperscript{[5-6]}. Negative ions can effectively reduce the pressure caused by computer operation\textsuperscript{[7]}. Chunyun Chen\textsuperscript{[8]} successfully prepared the anion powder with excellent dispersibility by sol-gel method, and successfully developed functional coatings containing different concentrations of negative ion powder. Their results showed that the functional material containing negative ion powder had certain antibacterial activity. At present, negative ion powder has been widely used in environmental protection, cigarette, paint, textile, water purification, health care and other fields.

Adding negative ion powder as a filler to the compound resin can make the compound resin have certain antibacterial properties, in order to reduce the occurrence of microleakage and secondary spasm after repair, and prolong the life of the prosthesis in the oral cavity, which has attracted the attention of many scholars at home and abroad.

2. Methods

2.1. Material and equipment

2.1.1. Material. The materials and reagents used in our experiments include anion powder (Self-processing of ultrafine powder in Shijiazhuang, Hebei Province), absolute ethyl alcohol (Rionlon Bohua Pharmaceutical & Chemical Co. Ltd., Tianjin Province), silane coupling agent (Tianjin Chemical Reagent Co., Ltd., N, N-dimethylformamide(DMF) (Lianlong Bohua Pharmaceutical Chemistry Co., Ltd., Tianjin Province), high purity deionized water (DW) (Millipore Company, USA) and DF light Curable Composite Resin (Kulzer GmbH).

2.1.2. Equipment. The equipment we used in our experiment included scanning electron microscope (JEOL, 5600LV, Japan), light curing machine (Densberg Company, USA), super centrifuge (Optima L-100XP, USA), microhardness tester (Hengyi precision instrument Co. Ltd. China) and fourier transform infrared spectroscopy (ATR-FTIR, IFS66V/S, Bruker).

2.2. Preparation of compound resin composite containing negative ion powder

In this experiment, the negative ion powder is dispersed into the resin matrix material by solution mixing method\textsuperscript{[9]}, and anhydrous ethanol is used as a solvent to add a negative ion powder. Due to the bonding strength between the base free filler and the organic matrix is weak, and the silane coupling agent can improve the adhesion strength and durability between the compound resin and the solid surface, however, in the mixing process, because of the small diameter of the inorganic filler particles, it is easy to agglomerate. In this experiment, mechanical agitation and ultrasonic dispersion were used to prevent particle agglomeration.

(1) Experimental group: The negative ion powder was added to the compound resin at a ratio of 1%, 2%, and 4%, respectively. Take 4g resin dissolve with anhydrous ether, add a drop of silane coupling agent, mix evenly with magnetic agitator and ultrasonic oscillation according to the proportion mentioned above. Finally, the ethyl ether was removed by rotating evaporator, and different proportion of negative ion powder compound resin was prepared.

(2) Control group: 4 g of a compound resin sample to which no negative ion powder was added was taken, and treated in the same manner as in the experimental group to prepare a compound resin containing no negative ion powder in the control group.

2.3. Scanning electron microscope (SEM)

The sample containing 0%, 1%, 2%, and 4% negative ion powder was irradiated vertically with a curing machine under a curing condition of 500 w/cm 2*40s, the sheet-like solid block was prepared, and the surface was polished to spray gold, and then the dispersion of the negative ion powder was observed under SEM.
2.4. FT-IR
Scan the KBr background curve, and then apply a thin layer of KB on the prepared compound resin in the dark, and test the infrared spectrum curve. After taking out, it was uniformly cured by a photocuring machine, and the infrared spectrum curve after curing was tested. In the N2 atmosphere during the test, the vacuum in the sample compartment and the optical chamber was less than 3*10^-5Pa. The measurement of the control group, 1%, 2%, and 4% compound resin was completed, the infrared curve was analyzed, and the curing rate of each group was calculated. The curing rate was calculated according to the following equation:

\[ RC\% = \left(1 - \frac{Ac}{Ao}\right) \times 100\% \]

Of which, Ac is the infrared 1640cm^-1 absorption peak spectrum after curing, and Ao is the absorption peak area of infrared 1640cm^-1 before curing.

2.5. Microhardness test
The hardness of anion containing compound resin materials was tested with reference to international standard (ISO 4049-1978) and China dental compound resin material standard (YY 91042-1999), each material was tested 3 times and its average value was taken.

Preparation of hardness specimen: Hardness test standard: 20mm×10mm×1mm; each group had 3 specimens.

Curing condition: 500w/m²×40s, vertical irradiation. Each side of the test piece was polished with 600 mesh water sandpaper, polished, and the test piece was stored in physiological saline at 37 °C for 24 h.

Hardness testing: The diamond quadrilateral pyramid was used as the indenter, the specimen was placed under the pressure head of the microhardness tester, and the microscope was adjusted until the specimen was clear under the microscope. Press down on the indenter and mark the length of the two crosshairs of the indentation through the eyepiece. Read the hardness value (HV) displayed by the instrument. Loading: 500mg, probe indentation time: 10s.

2.6. Data statistics
One-way ANOVA and T test were carried out by using SPSS10.0 statistical analysis system software.

3. Result and discussion

3.1. SEM observation
The results of the surface SEM of the compound resin containing 0%, 1%, 2%, and 4% negative ion powder are shown below (Figure 1). Figure 1A shows the absence of negative ion powder particles in the control group. In Figure 1B, 1C, and 1D, there are many cube-shaped negative ion powders that have been mixed into the compound resin, and are not agglomerated and dispersed well, which indicates that negative ion powder and compound resin are successfully combined by this method, and negative ion powder can be dispersed uniformly without agglomeration.

3.2. Curing rate result

3.2.1. Infra-red spectrogram. The two figures are Infra-red spectrogram of absorption peak area of infrared 1640cm⁻¹(Figure 2, Figure 3).
Figure 2. The absorption peak area of infrared 1640cm⁻¹ before curing.  
Figure 3. The infrared 1640cm⁻¹ absorption peak spectrum after curing.

3.2.2. Data processing. According to the above formulas, the curing rate of the composite resin is calculated as shown in the table.

| Negative ion powder content | Curing rate |
|----------------------------|-------------|
| Control                    | 20.3        |
| 1%                         | 24.2        |
| 2%                         | 23.7        |
| 4%                         | 23.9        |

At present, in the clinical use, blue light is mainly used as a light source to activate an initiator at a wavelength of about 400-500nm, so that the compound resin is intraorally cured, however, the solidification was not complete at the temperature in the mouth, and some of the monomers remained. It is reported that the curing rate of most resins ranges from 40% to 70%[10]. The degree of monomer conversion marks the curing degree of the resin and directly affects the physical properties of the resin, such as wear resistance, hardness, etc. In the polymerization process, the aliphatic C=C is opened and crosslinked into C-C, so the less the residual aliphatic C=C content after curing, the higher the degree of curing. The monomer conversion rate is high, the residual monomer is small, and the physical properties are high. The results showed that the cure rate of the experimental group was higher than that of the control group after adding negative ion powder, which indicated that negative ion powder had no effect on the curing rate of compound resin(Table 1). FTIR is a very effective and commonly used method to measure the conversion rate of compound resin. After the compound resin is cured, C=C is opened, and the peak area is relatively reduced in the infrared spectrum. The ratio of the peak area before and after curing can reflect the curing rate of the compound resin.
3.3. Surface microhardness result

![Image of microhardness test results]

The results showed that the microhardness of negative ion powder was significantly lower than that of control group after adding compound resin in different proportion, and the results were statistically significant compared with the control group (Figure 4). The decrease of microhardness increases with the increase of negative ion powder content[11].

4. Conclusion
In this experiment, compound resin containing negative ion powder was successfully prepared. Firstly, a mixture of different concentrations of negative ion powder was uniformly mixed into the compound resin by mechanical stirring dispersion and ultrasonic dispersion, and a silane coupling agent was added. The degree of dispersion was observed by SEM, and the curing rate and surface hardness were measured. The addition ratio of negative ion powder has ideal mechanical properties, but its antibacterial properties need to be further studied, and clinical trials are required to test its clinical application effect.

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