# Preparation and Applications of Polyamide 6/ IRM® Tooth Root Composite Filling Material

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## ABSTRACT

Intermediate restorative material (IRM®) is the most commonly used temporary filling material. This research mixed IRM with Polyamide 6 (Nylon 6) fibers, forming the Nylon/IRM tooth root composite filling materials. Tests such as setting time, degree of solubility, compressing strength and micro-leakage were carried out to examine the properties of the Nylon 6/

IRM® composite material. The result showed that there was no significant difference in the setting time and degree of solubility after adding the Nylon 6 fibers. The loading after the yielding point of the Nylon 6/IRM® was more than 250 N; micro-leakage was found on the 13th day.

**KEY WORDS**: Nylon 6 fibers, root end treatments, composite material.

## INTRODUCTION

Coming After Cancer and cardiovascular diseases, the dental caries has been identified as the third non-infection disease by the WHO, which also confirmed that there were more than five billion people bothered by the tooth decay problems. Because of the development of the sweets and delicate food, many patients are merely children living in the city. Oral cavities are common in Asia and South America.

The tooth root surgery is a medical treatment dealing with the dental pulp. It includes diagnoses and restoration. Diagnoses are to surgically find the sources of pain, pathological changes, the inflammation, and infections of the root end tissues. X-ray images are also examined for further prescriptions. The

tooth root surgery is to cut the infected tissue when the pathological change is found in the root and canine tooth tissues. After the pathological changes are removed, the tooth root is hold open by a reamer which is later applied to clean the inner and lateral side of the root. Rinsed by chloramine and perhydrol interchangeably, the root is then coated by the formalin cresol solution, the chloromycetin solution, and camphor, a common dental prescriptions. After sterilization, the root is filled with the temporary filling material. With the invention of convenient medical tools and the enlightenment of people's understanding over this matter, the surgical tooth root treatments nowadays is rare in the clinic [1].

A perfect filling material must meet certain requirements. Foremost, it should be physical stable to prevent shrinkage or swelling; furthermore, it must fit the cavity completely to block out microorganisms and toxicants. An ideal filling material, besides requiring sufficient working time, is an anti-carcinogen with characteristics like radio-resistance, bio-compability, antibacterial activity, moisture resistant and insolubility. Many vivo studies have been done to examine various filling materials, techniques and other factors such as hand dexterity. The root-end micro-leakage is frequently used.

It involves A. the dye leakage method, B. the fluid filtration method, C. the electrochemical method, D. the microorganism penetration method, and E. the radioisotope labeling method. Methods of the dye leakage, the fluid filtration and microorganism penetration are popular. Having a long history, the dye leakage method is the easiest. None of those methods can replicate the complicated root-end infection mechanism, so it is hard to tell which one is better [2-9].

The application of fibers has made a breakthrough in conventional attire manufacture. Nowadays, fibers are used to strengthen the mechanical property of production and enrich the functions of the composite. From 2004 to 2008, Lin et al. have utilized fiber materials in electromagnetic shielding effective products and medical dressings [10-26]. Fibers are frequently used to manufacture a multi-functional composite material. In the textile industry, this blending aims to expand the production line. In the dental treatment, the quality of the temporary filling material is primary. If the material fails to sustain occlusion, bacteria will make inroads into the tooth root and cause the secondary infection which will prolong the treatment. As a result, the temporary filling material must have high compressive strength. In this study,

the polyamide 6(Nylon 6) fibers was added into the temporary filling material to better the mechanical property of the material and prevent cavities caused by occlusion and bacteria crises. Finally, the influence of the addition of Nylon 6 fibers on the micro-leakage in temporary filling material was subsequently evaluated.

## EXPERIMENTAL

Zinc-eugenol (IRM®) was offered by Densply Amrican and the high strength Nylon 6 fiber was purchase by Formosa chemical and Fiber corporation. The Nylon 6 fibers was 64 mm length, 10 gf/D strength, 24.7 % elongation and 6.0 denier. Nylon 6 fiber was trimmed with different length (2, 4, 6, 8, 10 mm), after which it was mixed with IRM® to form the Nylon 6/IRM® composite material. The Nylon 6/ IRM® composite materials were prepared by mixing the IRM® with the 2 mm Nylon 6 fibers which were added in different weight ratios: 0.25, 0.5, 0.75, 1.0, and 1.25 wt%. This mixed gluey substance later was put in a mold of simulate tube and put to test its setting time (ADA#30), degree of solubility, compressing strength, micro-leakage to find out the best adding ratio. Besides, different lengths of the Nylon 6 fibers

(2, 4, 6, 8 and 10 mm) were also examined to have an appropriate fibers length with minimal micro-leakage.

**Solubility measurement**

The degree of solubility of test materials was determined by a modification method of the American Dental Association (ADA) specification #30 [7]. The materials were prepared in line with manufacturers' recommendations. After mixing, each substance was made into a small disc with a size of 20 mm × 1.5 mm by the use of a metal mould and two glass plates. Mixing and weighing of the samples were performed by a single operator at 23±2 ℃ and a relative humidity of 5-50 %. Six discs of each material were fabricated and tested. After fabrication, they were placed in 100 % humidity for 21 hours and then stored individually in glass bottles containing 50 ml of distilled water at 37 ℃. The specimens were removed from the water after 1 day. All discs were desiccated for 1 hour at 37 ℃. Each disc was then weighed to the nearest microgram. After weighing, each disc was replaced in the same glass bottle. The water in the bottles was neither changed nor added to during the test periods. The desiccation and weighing

procedure was performed at 1, 4, 7, 10 and 13 days.

**Micro-leakage measurement**

The micro-leakage test materials were determined according to the method recommended the American Dental Association (ADA) #30. It was putting in the dye solution of setting complete specimen. After picked up the specimen and cut it. And then used the stereoscope to observation the cross-section dyeing leakage in the specimen or not.

**Compressive strength measurement**

The specimen, 6 mm thick with 4 mm diameter, was first put in a distilled water, whose temperate was set at 37 ℃±1 ℃, for 24 hours in the water base, and then in another distilled water set at 23 ℃±1 ℃ for 15 minutes before the experiment. On the basic of American Dental Association (ADA) #30, this experiment used Instron5566 to run the test.

## RESULTS AND DISCUSSION

### Setting Time Test

As it shows in table 1, more Nylon 6 fibers lead to lesser setting time. The amount of fibers determined all. The Nylon 6/IRM® composite material consists of zinc powder, eugenol solvent and the Nylon 6 fibers. The surface of the Nylon 6 fibers absorbs solvent in the mixture; thus, the eugenol solvent, used to reactive with the zinc powder, was decreased simultaneously. Consequently, the volume of the eugenol solvent is lessened; the setting rate of Nylon 6/IRM® was more quickly; the setting time came sooner.

In Table 2, the longer the Nylon 6 fibers, the shorter the setting time. This was ascribed to the longer length of the Nylon 6 fiber, which caused higher capillarity, resulting in a greater amount of solvent attached onto the fibers' surface. Hence, the eugenol which interacted with zine-oxide was on the decrease, reducing the setting time comparatively.

The setting time was decreased (Table. 1 and 2) regardless of weight ratios and fibers lengths. This reduction was minor, though. Table 2 demonstrates the setting time of the Nylon 6/ IRM® composite material, made up of the heavies weight (1.25 wt%) and the most lengthy fibers (10 mm) was

345 ± 20 sec; however, the decrease barely influenced the working time. This reduction did not speed up the clinical procedure significantly.

**Compressive Strength Test**

Figure 1 presents the loading strain curve of the Nylon 6/ IRM® comprising of different Nylon 6 fibers weight ratios. Without adding the Nylon 6 fibers, the IRM® specimen crashed immediately in the test. Figure 2 demonstrates that the flaws, found on the surface and the inner side of the substance, indicate that the material's compressive strength was too weak to bear the loading. As the loading became heavier, the flaws were born to bear the increased burdens. When the loading was beyond the material's yielding point, the flaws were accordingly expanded to a state that the structure was deformed and destroyed. Figure 1 shows that the loading of sample without fibers, i.e., IRM sample, plummeted after it received the maximum loading.

Figure 1 shows that more Nylon 6 fibers gave rise to higher loading when the given loadings reached the yielding point. Figure 2 exhibits the specimen's shape after the compressive strain test. It was apparent that the specimen was compressed and deformed; however, wholeness was remained still. The Nylon 6/ IRM® composite material seemed to retain a more complete structure than

that without the Nylon 6 fibers.

The interface bonding between the Nylon 6 fibers and the filling material was good so that the Nylon 6 fibers were capable of dispersing the stress for the filling material. Hence, the addition of Nylon 6 fibers could reinforce the structure of the filling material, preventing the filling material from collapsing under compressive stress.

At the right side of the loading strain curve (Figure 1), there was a higher loading than that of the IRM® material.

Figure 3 demonstrates the loading strain curve of the Nylon 6/IRM® made up different weight ratios of the Nylon 6 fibers. When the loading was beyond the yielding point, the loading at the right side of the figure was higher as the used fibers grew longer. The longer the fibers, the better the property would be.

Figure 4 display the exposed fibers of the Nylon 6/IRM® . The thickness of sample was 6 mm with a 4 mm diameter. The fibers longer than 4 mm were likely to expose themselves out of the samples, as shown in Figure 4. The circled area in figure 4 was destroyed and might result in micro-leakage. Consequently, 4 mm long fibers were efficient to reduce the amount of

exposed fibers.

**Degree of Solubility Test**

Figure 5 and 6 show the degree of solubility of the Nylon 6/IRM® consisting of different weight or fiber lengths. All samples exhibited a solubility ranging from 0.3 to 0.5%. Added Nylon 6 fibers did not change the chemical property of the IRM® material. Both the IRM® and the Nylon 6 fibers were non-water-soluble substances. Consequently, the results of the degree of solubility varied little. The structure of the Nylon 6/ IRM® composite material was intact because the Nylon 6 fibers was non water soluble.–

**Micro-leakage Test**

Figure 7(a)-(e) display the micro -leakages of the IRM® materials on the $1^{st}$, $4^{th}$, $7^{th}$, $10^{th}$ and $13^{th}$ days. No micro-leakage was detected, so it proved that the IRM® material strengtheneded good tightness which guaranteed a non-micro-leakage enviornment where saliva was kept outside the IRM® material. A material without micro-leakage rejects not only saliva, but also bacteria that usually accompany saliva. Bacteria infection is likely to affect the

debridemented tooth root and prolongs the treatment.

Figure 8(a)-(e) present the micro-leakage of the Nylon 6/ IRM® material on the 1st, 4th, 7th, 10th and 13th day. There were no micro-leakages on the 1st, 4th, 7th and 10th day; however, significant micro-leakage was found on the 13th day. The water soluable dye travelled through the fibers by capillarity; thus, micro-leakage was occurred. Accoridng to the result, a risk-free course of treatment was 10 days if the Nylon 6/ IRM® composite material, consisting of the 1.25 wt% Nylon 6 in fibers length of 4 mm, was used as the filling material.

## CONCLUSION

According to the setting time test, the adding weight ratios and fiber lengths of the Nylon 6 fibers were efficient to shorter the settign time. The more the adding weights or the lengthier the fibers, the shorter the setting times were. This diminution did not affect the working time; thus, high tensile Nylon 6 fibers works.

The degree of solubility test exhibited that the high tensile Nylon 6 fibers did not change the IRM®'s solubility. In the compressing strength test, the

strain of the IRM® material was 20.1842 N. In the strain curve, the best adding weight ratio was 1.25 wt%. When the loadings reached the yielding point, the strain of the Nylon 6/IRM® material, consisting of 4 mm long fibers, was higher than 250 N, twice than that of the 2 mm fibers.

Micro-leakage was seen in the Nylon 6/ IRM® composite material; thus, the 4mm fibers was the perfect fibers length. In the test, there was a noticeable micro-leakage of the Nylon6/ IRM® compoiste tooth root filling material on the 13th day. It suggested that the Nylon 6/ IRM® composite material would be micro-leakaged in a long term; however, this composite material certainly bettered the IRM® 's compressive strain quickly.

This study successfully strengthened a material by adding fibers. This method is widely applied to enforce the tooth root or the tooth base. The comporessive strength and the strain of the tooth root filling material, IRM®, were improved because of the Nylon 6 fibers.

## Acknowledgement

We are highly grateful to the laboratory of fiber application and manufacturing of Feng Chia University for providing this research with testing equipments to complete this experiment.

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