



## EFFECT OF DIFFERENT TEMPERATURES ON THE PHYSICAL PROPERTIES OF DIFFERENT ROOT CANAL SEALERS – AN IN VITRO STUDY

### Dental Science

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### ABSTRACT

**Aim:** To evaluate effect of different temperatures on the physical properties like flow and setting time of resin based sealer, silicone based sealer and MTA based sealer.

**Materials and methods:** Three different root canal sealers i.e. Resin based, Silicone based, MTA based were tested to evaluate the flow and final setting time. 10 samples of each of the sealers were packed in stainless steel ring moulds, after exposing to different temperatures, setting time of these sealers were determined using Vicats needle apparatus. To test the flow of sealers 0.05 ml of the sealer was dispensed on to the glass slab, another glass slab along with weights were placed over it. The minor and major diameter of sealer were measured using digital calliper. Paired t-test and One way Anova test were used to analyse the data.

**Results:** Silicone based sealers had highest flow at normal temperatures whereas when the temperatures were raised to 140°C silicone based sealers showed least flow. Setting time at 37 °C for all 3 sealers remained unchanged. On increasing temperatures at 140°C, the setting time of Silicone based sealer was reduced compared to that of Resin based sealers while MTA based sealers remained unaffected.

**Conclusion:** In this study Resin based sealers and Silicone sealer showed changes in the physical properties (setting time and flow) at high temperatures. Flow of the sealers were also influenced at different temperatures whereas MTA based remained unaffected at all temperatures.

### KEYWORDS

Endodontic sealers, Flow, Setting time, resin based sealers, silicone based sealers, MTA based sealer.

### 1.INTRODUCTION

Endodontic sealers are used in the obturation of root canal systems to achieve a three dimensional fluid-tight seal throughout the canal including the apical foramen and canal irregularities and minor discrepancies between the dentinal wall of the root canal and the core filling material. An ideal root canal sealer should provide excellent seal when set, dimensional stability, sufficient setting time to ensure working time, insolubility against tissue fluids, proper adhesion with canal walls, and biocompatibility.

Therefore, sealers help prevent leakage, reduce the possibility of residual bacteria from the canal to invade the periapical tissues, and resolve the periapical lesion.<sup>1</sup>

Adequate flow ability allows for the filling of irregularities. High flow may result in apical extrusion, leading to injury of the periapical tissues because of the cytotoxicity of the sealers. Thus, the flow characteristics of endodontic sealers have attracted attention recently.

The commercially available sealers are categorised according to chemical components: zinc oxide eugenol based, calcium oxide based, resin based, glass ionomer based, silicone based and MTA based sealer. Epoxy resin based sealers were introduced in Endodontics by Schroeder and current modifications of original formula are widely used for root canal filling procedures.

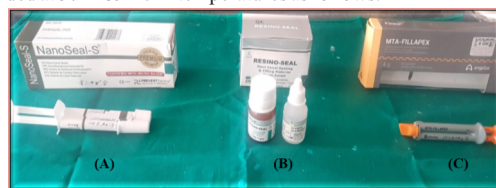
Studies on the effect of temperature on obturating materials have shown that techniques of gutta-percha placement involving heat in the root canal causes reversible physical changes in the gutta-percha without any apparent change in its chemical composition.<sup>2</sup>

With the new root canal filling sealer and the popularity of warm vertical compaction in practice (application of various temperatures in compaction), the effects of heat produced on these materials need further investigations. Thus this in-vitro study aimed to measure and evaluate the changes in the physical properties of three different root

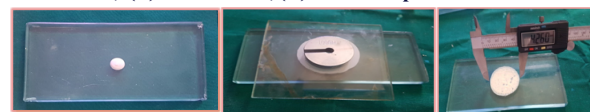
canal sealers at 37°C & 140°C temperatures.

### 2. MATERIALS AND METHODS

Three different root canal sealers were used as experimental materials viz, resin based: Resin-o-seal sealer (Ammdent), Silicone based: Nanoseal-S sealer (Prevest DenPro) and MTA based: MTA fillapex sealer (Angelus) were used as experimental materials. The physical properties like flow and setting time of these three sealers were recorded at 37°C & 140°C temperatures as follows.



**Fig.1: Three root canal sealers tested in the present study, (A) Nanoseal-S, (B) Resin-o-seal, (C) MTA Fillapex**

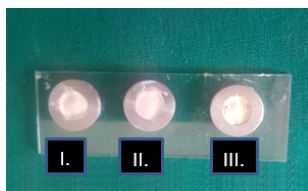


**I. 0.05ml sealer dispensed      II. Placement of 20 gms glass plate and 100gms weight over sealer      III. Diameter measurement using digital calliper**

**Fig.2 Method for measuring flow**

**FLOW (At 37°C):** Testing done with accordance to ISO Specification 6876/2001. A volume of 0.05mL mixed sealer was placed on the center of a glass plate by using a graduated disposable 3mL syringe. Three minutes later, a second glass plate weighing 20g and a 100g weight were placed centrally on top of the sealer. After 10 minutes from the

start of mixing, the load was removed, and the minimum and maximum diameters of the sample disks were measured by a digital calliper with a resolution of 0.01 mm (as shown in fig.2). If the disks were not uniformly circular (the maximum and minimum diameters were not within 1 mm), the test was repeated. Five tests were taken for each sealer, and the mean, was considered to be the flow of the material.



I. Sealers transferred in moulds before



II. Testing of samples using Vicats apparatus after exposing to heat



III. Indentations over samples after testing

**Fig.3 Method for measuring setting time**

#### SETTING TIME:

Testing was done in accordance to ADA specification no. 57 Resin based, Silicone based, MTA based sealers along with stainless steel ring molds with a inner diameter of 10 mm and a height of 2 mm were used. The molds were placed on the glass plate and then the materials were mixed and packed into them. The whole assembly of half samples was then transferred to an incubator (37°C, > 95% relative humidity). The other half samples were transferred to electric heating oven at 140°C.

The Vicats needle was lowered vertically onto the horizontal surface of the sealer and the setting time was identified as the point when the indenter needle failed to make an indentation (As shown in Fig 3). The materials were tested every 2 minutes depending on the setting time.

#### STATISTICAL ANALYSIS:

Statistical analysis was done, paired t-test and One-way Anova test was performed to determine setting time (Table 3) and flow (Table 1 and table 2) of the three sealers respectively. The significance level was set at  $p < 0.05$ .

**Table 1: One-way ANOVA Test: Comparison of flow of three sealers using One way Anova test (N= no. of readings per sealer, F= fratio)**

| Flow(mm) at 37°C     | N  | Mean  | SD    | F      | Df | P value                    |
|----------------------|----|-------|-------|--------|----|----------------------------|
| Resin based sealer   | 25 | 45.58 | 2.014 | 149.22 | 2  | < 0.00; Highly significant |
| Silicon based sealer | 25 | 54.39 | 2.304 |        |    |                            |
| MTA based sealer     | 25 | 49.32 | 0.714 |        |    |                            |
| Total                | 25 | 49.77 | 4.058 |        |    |                            |

**Table 2: Post-hoc test: Bonferroni test (Multiple pair-wise comparison)**

| (I) Groups  | (J) Groups   | Mean Difference (I-J) | Std. Error | P value |
|-------------|--------------|-----------------------|------------|---------|
| Resino Seal | Nanoseal S   | -8.81*                | 0.51307    | < 0.001 |
| Resino Seal | MTA Fillapex | -3.74*                | 0.51307    | < 0.001 |
| Nanoseal S  | MTA Fillapex | 5.07*                 | 0.51307    | < 0.001 |

\* indicates that the mean difference is significant at the 0.05 level

**Table 3: Paired t-test: Comparing the setting time of three sealers using paired t-test**

| Setting time (ST) (minutes) at 140°C | Reduction After 20 minutes | T   | P value           |
|--------------------------------------|----------------------------|-----|-------------------|
| Resin based sealer                   | 4.5                        | 8.0 | 0.02; Significant |
| Silicon based sealer                 | 0.5                        |     |                   |

#### RESULTS:

Setting time at 37°C for all 3 sealers remained unchanged. On increasing temperatures at 140°C, the setting time of silicone-based sealer was reduced compared to that of Resin based sealer while MTA based sealer remained unaffected. Flow of silicone-based sealer was highest, while Resin based sealers showed least flow at 37°C w.r.t. table 5.

Silicone based sealer showed highest flow of 54.34mm followed by MTA based sealer which showed 45.59mm flow. Among these three sealers Resin based sealer showed least flow of 45.59mm at 37°C w.r.t. table 5.

**Table 4: Setting Time**

|         | Resin based sealer |         | Silicone based sealer |         | MTA based sealer |       |
|---------|--------------------|---------|-----------------------|---------|------------------|-------|
| MINUTES | 37°C               | 140°C   | 37°C                  | 140°C   | 37°C             | 140°C |
| 10 mins | NA                 | 5-10min | NA                    | 0-5 min | NA               | NA    |
| 20 mins | NA                 | 0-5 min | NA                    | 0-2 min | NA               | NA    |
| 30 mins | NA                 | 0 min   | NA                    | 0 min   | NA               | NA    |

Setting time (in minutes) of three sealers at the intervals of 10 minutes each, tested for 30 minutes and above when exposed to 37°C and 140°C respectively.

Where NA=not affected.

**Table 5: Flow Mean readings (in mm) of the flow for three sealers at normal temperatures.**

|                       | AT 37°C (Mean in mm) |
|-----------------------|----------------------|
| Resin based sealer    | 45.59                |
| Silicone based sealer | 54.34                |
| MTA based sealer      | 49.31                |

#### DISCUSSION:

The chemical composition of root canal sealers that are used in close contact with periapical tissues is a predictive factor to understand their physical, chemical and biological properties.<sup>3</sup> However several studies have shown that heat does affect the physical properties and chemical composition of root canal sealers. It is well-known that the vertical compaction technique with warm gutta-percha produces consistently dense, dimensionally stable, 3-dimensional root canal fillings and has been developed to fill canal irregularities more effectively than is possible with cold gutta-percha.<sup>4</sup> Although the warm filling technique improves the compaction of gutta-percha into root canals, it is still necessary to use a sealer because any small voids between the gutta-percha and canal wall irregularities or canal fins have to be filled. It has been reported that warm gutta-percha root fillings without sealer leak more than those with sealer.<sup>5</sup> In the warm compaction technique, most thermoplasticized systems are operated at 200°C, and safety considerations dictate a heating duration of no more than 4 seconds.<sup>6</sup> A number of studies have investigated the actual temperatures of pluggers in thermoplasticized systems and found that they are at 50°C–126°C, which is much lower than that set on the LCD display.<sup>7-9</sup> According to the findings of our previous study, the actual temperatures of continuous-wave pluggers are below the set temperature 200°C, varying from 112°C–199°C (median = 140°C). When thermoplasticized systems operated at 200°C, the actual median temperature of pluggers was 140°C (112°C–199°C). Therefore, in the present study, a temperature of 140°C was set to evaluate the effect of a high temperature on sealers when warm vertical compaction was applied. In other similar studies, physical changes of sealers when heat was applied were observed. According to the actual temperature of System B pluggers (Analytic Technology, Redmond, WA), 100°C was used as the experiment condition in some studies.<sup>8-10</sup>

The setting time is dependent on the constituent components, their particle size, the ambient temperature, and the relative humidity.<sup>11-13</sup> Clinical convenience demands that it must be long enough to allow the placement and adaptation of root filling if necessary.<sup>14</sup>

The flow of endodontic sealers have effect on obturation of accessory canals and micro-spaces between master and accessory cones. Various factors such as composition, shear rate, particle size, temperature, and time from mixing are related to the flowability as well as setting time of sealers. All materials of the present study were in accordance with the ANSI/ADA specification no 57. The flow of endodontic materials can be affected by the geometry of the delivery system, the geometry of the root canal, and the physical conditions within the canal. Temperature selected was 37°C because it is mouth temperature. ISO standard for flow of endodontic sealers does not specify a temperature for the procedure, but it is an important factor in determining and comparing flow in materials.<sup>15</sup>

With novel endodontic sealers being successively developed and commercialized by manufacturers, it has become important for the clinician to understand the physicochemical properties of endodontic sealers. The properties of endodontic sealers, which are mainly determined by the type and proportions of the main components, can enable them to function adequately under clinical conditions. Laboratory studies on the physicochemical properties could contribute to a better understanding of the clinical behavior and handling performance of endodontic sealers. Recently, new endodontic materials have been developed that are based on the physicochemical properties of bioceramic cements in an attempt to develop a biocompatible sealer with ideal physical, chemical, mechanical, and biological properties. MTA based sealer is one of these recently developed endodontic sealers.<sup>15-17</sup>

According to the manufacturer's information, MTA Fillapex is composed of salicylate resin, resin diluent, natural resin, bismuth oxide as a radiopacifying agent, silica nanoparticles, MTA, and pigments. However, the details about the composition of the natural resin, pigments, and diluents are unknown. It is therefore necessary to investigate the physicochemical properties of the new endodontic sealers with the combination of the resins and other constituents.

Rheological properties include loss modulus ( $G''$ ) storage modulus ( $G'$ ), loss factor ( $\tan \delta$ ), and complex viscosity ( $\eta^*$ ).  $G'$  is a measure of stored energy and represents the elastic component of the material.  $G''$  represents the viscosity of the materials and is a measure of the energy lost as heat. Temperature 37°C (mouth temperature) was selected. ISO standard for flow of endodontic sealers does not specify a temperature for the procedure, but it is an important factor in determining and comparing flow in materials. MTA Fillapex which is calcium salicylate-based resin cement revealed  $G''$  greater than  $G'$  that attributes to gel-like viscoelastic behavior. It exhibited a crossover region in which the  $G''$  crossed the  $G'$  at both temperature conditions. This indicated, setting down behavior of MTA Fillapex at lower frequencies, and a change in molecular structure, a prospective structural breakdown beyond crossover frequency, which may be due to complexation type of auto catalytic reaction.<sup>18</sup>

The  $G'$  of Resin based sealer was higher than its  $G''$  over the entire experimental frequency range at both temperatures. The results indicated a dominant elastic solid-like behavior of this sealer. These findings may be due to epoxy-amine addition reaction that resulted to form a physical network in the structure of this sealer compared to other sealers.<sup>18</sup>

Among the clinically available root canal sealers, epoxy resin based sealers are widely used for root canal filling due to their resorption resistance and dimensional stability. Most recently introduced bioceramic based materials have attractive physical, chemical, mechanical and biological properties.

The International Organization for Standardization (ISO) 6876-2002/12 and the American Dental Association (ADA) specification no. 57 for endodontic sealers use the measurement of the diameter of material flowing between two glass plates under a specified weight for a specified time. These tests are preliminary tests and do not require measurement of viscosity which is a quantitative parameter for the evaluation of rheological properties of endodontic sealers and may help to achieve an ideal flow pattern. Rheological studies are useful to investigate the time-dependent viscoelasticity of the materials that are related to the working and setting times. Their measurement could give a more accurate indication of when the material is changing from liquid-like behaviour to solid-like behavior and how this affects their handling and sealing properties. Setting time is also important to

provide adequate working time and proper consistency enough to fill the root canal system completely. Setting times of evaluated sealers in this study were different from that given by the manufacture on influence of heat. In the present study, the temperature was increased from 37°C to 140°C and was tested according to ADA specification no. 57.<sup>18</sup>

Indentation in situ showed that the sealers had set because no indentation marks were left on the material surface. Resin based sealer showed higher setting time at 140°C as compared to silicone based sealer. MTA based sealer was not set in humid incubator condition and at higher temperatures of electric oven. When resin based sealer was heated, the setting time was reduced, as previously reported. It was suggested that this reduction was because of the loss of amine groups, which could accelerate the reaction. The reduction in the setting time of silicone based sealer (at 140°C) would not allow enough operating time for practitioners.<sup>5</sup>

An acceptable flow within the working time is important for any endodontic sealer to reach and seal the apical foramen and lateral dentinal wall irregularities. The composition, particle size, shear rate, temperature, and time from mixing are the main factors related to the flow characteristics of sealers. In accordance with the ISO 6876/2001 specifications the diameter of a film of sealer between 2 glass plates were used to evaluate the flow, which is related. The tested endodontic sealers are pseudoplastic, so that their viscosity is reduced and flow is increased when the shear rate increases during compaction to viscosity but easier to measure. Viscosity is a quantitative parameter for the evaluation of rheological properties of endodontic sealers and may help to achieve an ideal flow pattern.<sup>19</sup> Several studies have made an effort to examine the rheological properties of endodontic sealers by using a capillary rheometer<sup>19</sup> or extrusion through a bore<sup>20</sup>. However, to some extent, they are still indirect methods related to the viscosity measurements and do not provide the viscosity of sealers. The tested endodontic sealers are pseudoplastic, so that their viscosity is reduced and flow is increased when the shear rate increases during compaction. Pseudoplastic materials, particularly if they have low initial viscosities, will benefit from rapid placement and display a more rapid flow. On the other hand, an excessive flow rate increases the probability of extrusion into periodontal tissues. According to ISO specification, the sealers should have a diameter not less than 20 mm at flow test<sup>22</sup>. All the sealers tested in this study having flow above this value. The flow value of MTA Fillapex was higher to the value obtained by Silva et al. (31.09)<sup>23</sup>, Vitti RP et al. (29.04)<sup>24</sup>, Lee JK et al. (34.13)<sup>22</sup>. The manufacturer of MTA Fillapex does not provide any information about the composition ratio. A high resin/MTA ratio may be one of the reasons why a high flow rate occurs.<sup>22</sup>

## CONCLUSION:

Within the limitation of the current study Resin-O- Seal sealers and Nanoseal-S sealer showed changes in the physical properties (setting time and flow) at high temperatures. Setting time at 37 °C for all 3 sealers remained unchanged. MTA Fillapex remained unaffected at 37°C as well as at 140°C. Among these three sealers Resin-o-seal owed least flow of 45.59mm at 37°C.

However significant reduction in the setting time and flow of Nanoseal-S were found compared to Resin-O-Seal, which could negatively affect the performance of clinical practice and quality of obturation in warm vertical compaction technique.

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