



## The flow of two zinc oxide-eugenol-based endodontic sealers

### Napon tečenja dva cink-oksidi eugenolna endodontska silera

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#### Abstract

**Background/Aim.** Endodontic sealers (ES) for obturation are usually prepared with a slight variation of their components both on purpose or unintentionally. Considering that fact, as well as a frequent use of compaction techniques with the applied force to gutta-percha and ES of 1–3 kg, the aim of this study was to investigate the flow of two zinc-oxide eugenol ES in regard to the applied force and a variation of sealer's components. **Methods.** The experimental group samples of both ES were prepared according to the manufacturer's instructions, applied between pair of glass slabs and loaded by weights of 1 and 2 kg, respectively (American National Standard, Specification No. 57). Some samples of one ES were prepared as thick consistency with 10% more powder and some as thin mixture with 10% less powder than the standard prescription. These samples had been exposed to the load of 2 kg. The control group included samples of both ES prepared as standard prescription but exposed to the weight of one glass slab only. The spread ES appeared as a regular circle 10 min upon mixing and weighting. Measuring of the circle diameter was done by an orthodontic ruler. The flow of the used ES was considered the function of its spread

diameter. **Results.** Application of 1 vs 2 kg load for both regularly mixed sealers in the scope of disk diameter (flow) was statistically insignificant ( $p > 0.05$ ). This means that the stated null hypothesis that there would be no significant difference in flow rate among the regularly mixed sealers at the level of  $\alpha = 0.05$  is accepted. The findings about difference in the disk diameter in regard to mixing variation of Endomethasone indicate that the null hypothesis that there would be no significant difference in flow rate between the regular and thick mixed mass at the level of  $\alpha = 0.05$  is accepted. In the comparison of regular and thin mix a significant difference was noted and the null hypothesis is rejected ( $p < 0.01$ ). The control group results displayed Roth 801 as less viscous than Endomethasone sealer ( $p < 0.01$ ). **Conclusion.** Application of 1 or 2 kg pressure on the samples of both exposed sealers does not significantly affect the flow values as well as comparison of the regular to thick consistency of Endomethasone while comparison of its regular to thin mass shows a significant difference.

**Key words:** root canal filling materials; zinc oxide-eugenol cement; rheology; viscosity.

#### Apstrakt

**Uvod/Cilj.** Endodontski sileri (ES) za opturaciju kanala korena zuba često se u praksi pripremaju sa varijacijama svojih komponenti. Uzimajući u obzir ovo, kao i činjenicu da se sve češće koriste metode kompakcije gutaperke i ES sa primenjenim pritiscima od 10 do 30 N, cilj ovog rada bio je ispitivanje napona tečenja (*flow*) dva ES na bazi cink-oksidi eugenolnih silera. U tom smislu je planirano ispitivanje promene napona tečenja kod ES sa i bez primene opterećenja kao i sa minimalnim odstupanjima gustine materijala od regularno zamešane preskripcije (gušća i ređa konzistencija). **Metode.** U eksperimentalnoj grupi uzorci dva cink-oksidi eugenolna ES pripremljeni su prema uputstvu proizvođača, a zatim nanešeni između staklenih pločica i opterećeni tegovima od 1 i 2 kg (American National Standard, Specification No.57). Deo uzorka jednog silera bio je pripremljen kao gušća i ređa konzistencija (mešavina sa  $\pm 10\%$  praha od preporučene razmere) izloženih sili od 2 kg. Kontrolnu grupu činili su uzorci oba ES zamešanih prema uputstvu proizvođača bez optere-

ćenja, izloženi samo težini jedne staklene pločice. Veličina napona tečenja posmatrana je u funkciji prečnika razlivenog silera kao parametra napona tečenja između para pločica. **Rezultati.** Poređenjem uzoraka (prečnika razlivenih silera) opterećenih sa 1 prema 2 kg kod oba materijala, nađena je statistički neznačajna razlika ( $p > 0,05$ ). Nalazi u vezi prečnika razlivenih silera u pogledu varijacije  $\pm 10\%$  praha kod ES *Endomethasone N* ukazuju na to da ne postoji značajna razlika u naponu tečenja između standardno i gušće zamešane mase ( $p > 0,05$ ), dok je razlika bila značajna poređenjem uzoraka standardno zamešane mase prema onima sa ređom konzistencijom ( $p < 0,01$ ). **Zaključak.** Promena sile sa 1 kg na 2 kg kod uzoraka oba silera ne utiče značajno na napon tečenja kao ni poređenje standardno i gušće zamešanog *Endomethasone*, dok je poređenjem njegove standardne i ređe zamešane mase utvrđena značajna razlika.

**Ključne reči:** zub, materijali za punjenje korenskog kanala; cink-oksidi eugenol pasta; reologija; viskoznost.

## Introduction

The main function that a root canal sealer and gutta-percha should meet during obturation are lubrication and setting the master and auxiliary gutta-percha cones acting as intermediary and sticky-adhesive substance in a labyrinthine depulped space. The outcome of endodontic therapy might depend on sealers' properties whether they are biological, chemical or physical ones. The flow of endodontic sealer (ES) is affected by its viscosity as well as temperature and humidity. By the way, it is obvious that sealer's flow depends on the shape, width and taperness of the root canal.

An adequate consistency is required whether to use a paste carrier (Lentulo spiral filler) or soaked gutta-percha point. The endodontists often adjust the powder/liquid ratio to the appropriate consistency of the sealer, usually in eugenate sealer materials. The most desirable consistency should be chosen considering the 2 aims: not to overfill the apical canal portion when thicker consistency is required (wide open apical foramen, unfinished root growth), and on the contrary, weak consistency, is desirable when last millimeter of canal is not to be well obturated, i.e. strongly curved/narrow canals.

Some authors advocate for cleaning of smear layer as the important condition for ES flow and its penetration into the dentine tubules<sup>1</sup>. On the other hand, during the use of compaction techniques by the instruments and devices for

investigation of ES flow was done through notification of microleakage into the lateral canals or tubules<sup>13-17</sup>. The aspect of contact angles at 4 ES points out the correlation on their flow properties<sup>18</sup>.

Japanese authors<sup>19</sup> compared the two testing device values (vertical plate and two-plate system) of the flow on the same sealers. Extrusion viscometer<sup>20</sup> or free extrusion of ES through the bore<sup>21</sup> has been used for research on the rheological characteristic.

Considering the aforementioned, the aim of this study was to investigate the rheology features by influence of powder: liquid ratio and the two forces applied to the zinc-oxide eugenol (ZOE) ES. The first null hypothesis was that there would be no significant difference in flow rate among the regularly prepared sealers at the level of  $\alpha = 0.05$  and regardless the applied load of 1 and 2 kg at the level of  $\alpha = 0.05$ . The second null hypothesis was that there would be no significant difference in flow regardless the sealer consistency and in comparison to regularly prepared mix considering the level of  $\alpha = 0.05$ .

## Methods

### *The root canal sealers*

The two ZOE preparations as ES were tested for the study whose approximate contents according to the manufacturer are given in Table 1<sup>22,23</sup>.

**Table 1**

**The approximate composition /main ingredients/ of the used zinc oxide-eugenol (ZOE) endodontic sealers**

ZOE endodontic sealers	Ingredients
Endomethasone N (Septodont) powder	zinc-oxide, magnesium stearate, thymol iodide, barium sulphate, hydrocortisone acetate, excipients liquid: eugenol, excipients <sup>22</sup>
Roth 801 (Roth Inter Limit.) powder	zinc-oxide, staybelite resin, bismuth subcarbonate, barium sulphate, sodium borate anhydrous liquid: pure eugenol <sup>23</sup>

obturation mass, sealer or gutta-percha cones pressing, high values of exposed pressure act as hydrodynamic pump to the root-canal walls. A result of compaction forces should be visible in filling all the canal irregularities, accessory ones as well as apical delta due to high exposed values of lateral and vertical forces. Some authors apply the force of 2 kg (~19.6 N) imitating clinical compaction stress, while the others use zinc-oxide eugenol ES and real clinical obturation force of 10, 15, 20 and the 25 N in studies on the quality of apical seal<sup>2-3</sup>. Their exams were based on the study where the average manual force during obturation ranged 10-30 N among eight endodontists<sup>4</sup>. Application of heat in some obturation techniques may also influence the flow characteristic of a sealer<sup>5</sup>.

Various devices have been used in evaluation the rheological properties of ES. This might be the reason for not having any important laws and conclusions about flow properties of sealers. In the study on temperature influence on sealer custom-made capillary rheometer<sup>5,6</sup>, and cone-and-plate geometry were used<sup>5</sup>.

ES flow rate has been studied by some investigators using a vertical glass plate<sup>7</sup> or a 2-plate system<sup>5,8-12</sup>. Some

### *The study groups*

The study involved the experimental and the control group.

The experimental group involved the samples of a regular and varied mixture of the two aforementioned ZOE ES (Table 1). Regular mixtures of Endomethasone (12 samples) and Roth 801 (10 samples) were subjected to the load of 1 kg and 11 samples per each sealer to the load of 2 kg. The varied Endomethasone samples were prepared as thick and thin consistency (11 samples of each) and exposed to the load of 2 kg.

The control group included 3 samples of each used sealer regularly prepared. They were exposed to the weight of only one mixing slab (100 g).

The load exposure time was 10 min for all samples in the experimental and control groups.

### *The components ratio*

Endomethasone N was prepared as regular prescription with ratio: one spoonful of powder to two drops of liquid<sup>22</sup>. Thick consistency contained 10% more powder (by weight)

than regular mix. Thin consistency had 10% less powder than regular mix. The reason to choose so minimal deviation of standard proportion ( $\pm 10\%$  of powder) was reality of clinical situation where many times ES is prepared by such a varied proportion unintentionally or intentionally, as well as that no up-to-date literature data appeared of such study concept.

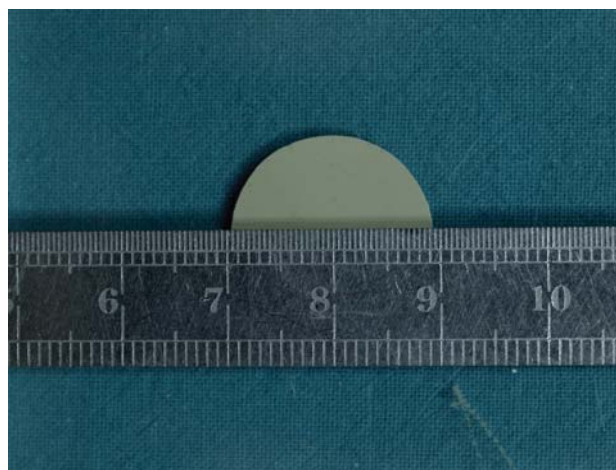
Roth's 801 sealer was prepared as regular prescription to the consistency of petrolatum gel by the powder liquid ratio of 0.13 g : 0.03 g<sup>21</sup>.

The adjustment of components was done by digital scale device with accuracy of 0.001g (Mettler PE 360, Germany).

*The protocol and the experimental device for the experimental and control groups*

The experiment methodology was based mostly on the 2-glass-plate geometry system (ADA specification No 57).

The ES were mixed according to the directions of a corresponding manufacturer and varied for Endomethasone samples<sup>24</sup>. The same amount of sealer (0.06 mL) was placed immediately after mixing by graduated syringe to the center of glide mixing plate and spread for 1 min by dental probe forming the circle of approximately 10 mm diameter. Another glass plate weighing 100 g was then gently placed over the first 3 min after initiation of mixing. An extra load of 1 and 2 kg was added for the samples in the experimental group. A 2-glass-plate system was then fixed laterally to prevent minimal moving. All 4 brinks of the 2-glass-plate unit



**Fig.1 – The disk diameter of spread sealer measured by orthodontic ruler**

tions of two components in Endomethasone N samples as well as for comparison between Endomethasone N and Roth 801 viscosity values for regular mix and load of 2 kg.

**Results**

The mean disk diameter values of spreading regular and varied prepared mixed mass of sealers upon exposing the load of 1 and 2 kg are presented in Table 2 for the experimental and in Table 3 for the control group.

**Table 2**  
**The disk diameter mean and coefficient of variation CV (%) values of spread regular and varied mixtures of sealers between the glass slabs upon exposing to the extra load of 1 and 2 kg (experimental group)**

Sealer	Mixtures			
	Regular prepared		Varied	
	1kg	2kg	thick (2kg)	thin (2kg)
	$\bar{x}$ (CV)	$\bar{x}$ (CV)	$\bar{x}$ (CV)	$\bar{x}$ (CV)
Endomethasone N	21.9 (8.5)	24.1 (8.7)	25.0 (11.6)	21.7 (9.1)
Roth 801	29.6 (12.0)	32.8 (17.7)	-----	-----

were fixed by ten tangentially placed 4 cylindrical metal weights of 1 kg. Measurements were done at the stable lab temperature of 22 °C and 65% humidity.

There was no load application except the weight of mixing slab of around 100 g in the control group samples.

*The measuring*

Measuring of values of the two perpendicular diameters were done in both experimental and control group 10 min after sealer application by the help of an orthodontic ruler of 0.5 mm raster and error of 0.025 mm (Figure 1). The values were recorded as the average estimation of two measured diameters summing the maximal and minimal values by the help of 4 × magnifying glass. The sample was discarded if the difference in the two recorded diameters per sample was more than 1 mm.

The Student's t-test was used for recording the differences in disk diameters among experimental samples in regard to the applied pressures (weight), in the cases of varia-

**Table 3**  
**The disk diameter and coefficient of variation (CV) values of spread sealer mass without extra load (control group)**

Sealer	Mean diameter (mm)	CV (%)
Endomethasone N	15.7	8.2
Roth 801	22.4	2.5

The difference in disk diameters of the mixed mass spread by exposing the load of 1 kg weight vs. 2 kg for both regularly prepared sealer mixture was not statistically significant ( $p > 0.05$ ).

A significant statistical difference in disk diameters values was found in comparison of Endomethasone N and Roth 801 sealer when regularly mixed and used 1 or 2 kg load ( $p < 0.01$ ).

Comparison of disk diameters values for 2 kg load of the regular and thick Endomethasone N preparation failed to show statistically significant difference ( $p > 0.05$ ).

A statistically significant difference in disk diameter was found in comparison of the regular Endomethasone N mixture to the thin mixed mass ( $p < 0.01$ ), as well as in thick vs thin Endomethasone N mixed mass ( $p < 0.01$ ).

The disk diameters in the control group within each sealer of no extra load were recorded as vary near values for both materials, thus provided the reliable parameters for statistical analysis (Table 3). Those mean values of disk diameters point out that the load of only one glass plate to exposed Roth 801 as sealer give provide statistically significantly bigger diameters than Endomethasone N ( $p < 0.01$ ) cases.

## Discussion

The obtained coefficient of variation (CV) values for samples in this study for both experimental and control group were far below 30%, *ie* in the ranges of 8.5%–17.7% and 2.5%–8.2%, respectively, what characterized them as statistically homogenous groups very suitable for a precise statistical analysis.

In this study two glass plates were used on the same way as Grossman did in his 1976 study<sup>25</sup>. The reason to choose this method is its simplicity as well as the presence of comparable literature data. The flow investigation under ISO specifications<sup>26</sup> on ZOE endodontic sealers (Canals, Showa) and sealer with other ingredients displayed diameter values higher than 20 mm (39.2–46.2 mm) thus satisfied ISO requirements<sup>19</sup>. A study on 3 sealer flow in a 2-plate system under ADA conditions exposed mean diameter values in the range of 32.7–37.9 mm with the highest values for ZOE sealer<sup>11</sup>. In the flow study of Endomethasone authors did not required the ADA specification No. 57, although of limited value (20 mm disk diameter) and ISO standard by the value of only 11 mm diameter<sup>13</sup>. The present study results satisfied ADA specification No. 57 of sealer with the mean diameter values higher than 20 mm (21.9–32.8 mm).

One can say that all aforementioned results were in the range of  $d \geq 20$  mm. The variation might be explained by a slightly performed deviation of the experimental conditions such as weight of a glass slab (30, 80, 100, 120, 450 or 500 gr), or extra load (1, 2 or 2.5 kg) the amount of the applied sealer on the plate (0.05, 0.06, 0.1 or 0.5 mL) and load exposure time (30 sec, 7 or 10 min).

In this study the comparison of Endomethasone N thick *versus* thin mix by glass plates revealed the significant difference in disk diameters. The difference of powder saturation between these two consistencies was around 20%. In comparison of the two ZOE ES with the deviation of around 30% of median consistency French authors noted the similar results<sup>12</sup>.

Although some authors used the powder increase of 50% in Grossman ZOE ES they did not obtain a significant difference in diameter values among thick and thin mix, as well as to Tubliseal ZOE ES, most probably due to negligence of both ADA and ISO specification. However, it is amazing that their study noted disk diameters larger than 20 mm for all sealers thus required the ADA conditions<sup>6</sup>!

The hypothesis that the flow is comparable with the penetration degree into dentinal tubules checked by SEM stated the authors who noted the ZOE Pulp Canal Sealer as low potential sealer in regard to resin-based sealer<sup>16</sup>.

The compaction force of 1.0 kg was applied in the study to imitate the Schilder plugger for compaction during obturation in test of various viscosity mixture<sup>27</sup>. According to the noted force values during compaction in the range of 8–35 N<sup>2,3</sup> the aim of this study was to compare those authors' results themselves. This is the reason to chose the load for of 10.0 and 20.0 N in this study.

An increase in intracanal pressure during the rise in sealer viscosity is noted both with more or less thick consistency<sup>27</sup>. This might be of high importance in thin roots due to the possible fracture<sup>28</sup>.

It is sometimes very difficult to make the accurate proportion of the powder and liquid or two-paste system because double-syringe or accurate spoonful or bottle for all brands of ES are sometimes missing. That is another reason to believe in unintentional variation of ingredients in the amount of  $\pm 10\%$  of one component during preparation. It is questionable if a slight variation of one component might significantly influence the rise or decrease in sealer flow and thus cause an unwanted change in planned clinical consistency up to the concerned clinical endodontic situation. The variation of sealer consistency was applied in this experiment due to the recommendation of the Endomethasone N manufacturer allowing regular ratio of 1 : 2 of powder and liquid with deviation of around  $\pm 50\%$ <sup>22</sup>. Actually, the manufacturer allows the mixing variation in the sense of thicker and thinner consistency depending on the clinical situation. Using the variations in powder of Endomethasone N of only  $\pm 10\%$  (clinical approved mixtur) and obtaining only a limited influence of the sealer's rheology, this study is characterized as the novelty in the literature data. The result of French authors about the influence of powder variation of around  $\pm 30\%$ – $50\%$  to the rheology of the two ZOE ES, Pulp Canal sealer and Cortisomol revealed a significant flow change<sup>12</sup>. Some authors obtained statistically significant differences in flow parameters comparing the viscosity of the mixture much thicker than much thinner ( $\pm 10\%$  of powder) and than regular ZOE sealer mix<sup>5</sup>.

The noted flow rate in vertical glass plate experiment of two ZOE sealers, Endomethasone and Procosol, revealed significant difference where both of them were exposed to significantly lower flow than resin-based and  $\text{Ca}(\text{OH})_2$  filler materials<sup>7</sup>.

Sealer extrusion through the bore did not showed significant difference of flow between two ZOE sealers Roth 801 and Tubliseal EWT<sup>21</sup>.

Although this study showed no influence of gutta-percha on the flow of obturation mass, it was shown that the flow of filling material such as ZOE ES depends on gutta-percha flow. It can be explained by the influence of the chemical compounds of gutta-percha cone that vary in different brands<sup>29</sup> what is advised to be studied in the next research.

Observing literature data sometimes presented as heterogeneous rheological characteristic of the ES obtained for the same materials but under slightly or largely changed conditions, point out to the need of strict following the standards in order to compare the results of investigators worldwide.

### Conclusion

Application of 1 kg *versus* 2 kg load for both regularly mixed sealers in the scope of the obtained disk diameter (flow) was statistically insignificant ( $p > 0.05$ ).

A significant statistical difference in disk diameters values (flow) was found in comparison of Endomethasone and Roth's 801 sealers both regularly mixed for application of 1 kg or 2 kg load ( $p < 0.01$ ).

The obtained difference in disk diameter in mixing variation of Endomethasone N and 2 kg load points out statistical insignificance in flow rate between regular and thick mixtures ( $p > 0.05$ ). A significant difference was found in comparison of regular and thin mixtures by the load of 2 kg ( $p > 0.01$ ).

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