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Flow, Film thickness and Setting Time Properties of Three Mineral Trioxide Aggregate Based Bio-ceramic Sealers: An in Vitro Study

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ABSTRACT
Background: The mineral trioxide aggregate (MTA) based bio-ceramic (MTA-BCS) has become quite well known for its top-notch amenities. The quality of the obturation procedure is influenced by the composition and handling characteristics of the root canal sealers. Flow, film thickness and setting time are the most important handling properties as they affected the ability of the sealers to enter narrow and irregular spaces.
Objective: The aim of the current study was to evaluate 3 commercially available MTA-BCS sealers regarding flow, film thickness and setting time.
Materials and methods: Three commercially available MTA based sealers were used in this study. Samples were divided into three groups; **Group I** (MTA fillapex), **Group II** (Ceramoseal HBC) and **Group III** (Endoseal MTA). Flow, film thickness, initial and final setting time were tested according to ISO 6876 standards.
Results: For the flow, test Group I showed the highest mean value (26.50±0.50 mm) and the lowest statistically significant mean value was Group II (22.59±1.94 mm). For the film thickness, the highest statistically significant mean value was recorded in Group II (29.4±4.72 µm) while the lowest mean value was recorded in Group III (15±3.53µm). For initial setting time, the highest statistically significant mean value was recorded in Group I (858.51±11.82 min). The lowest statistically significant mean value was recorded in Group III (35.2±2.86 min). For final setting time, group I was unable to set up to one month. Group III was the shortest statistically significant mean value (150.4±3.44 min) compared to Group II (832±6.78 min).
Conclusion: Within the limitation of this study, it can be concluded that all the tested sealers fulfilled the requirements of ISO specifications no 6876 for flow and film thickness. Regarding setting time, only Group II fulfilled their manufacturer declaration for initial setting as stated by ISO.
Keywords: Bio-ceramic sealer, Calcium silicate sealer, Ceramoseal HBC, Endoseal MTA, MTA fillapex sealer.

INTRODUCTION

To create a fluid-proof seal throughout the root canal system, endodontic sealers are utilised. Sealants stop leaks, lessen the chance that lingering germs from the canal may infiltrate the periapical tissues, and heal the periapical lesion [(1)]. Grossman claims that the optimum root canal sealers should produce a good seal, offer good adhesion, be radio opaque, and not discolour the tooth structure. They should also be insoluble in tissue fluids, easily mixed and injected into the canals, and dimensionally stable. Sealants should give adequate working and setting times, be bactericidal or bacteriostatic, and not irritate periradicular tissues [(2)]. Until now, none of commercially available endodontic sealers fulfil all these ideal requirements [(3)].

Zinc oxide eugenol, calcium hydroxide-containing, resin-based, glass-ionomer-based and bio-ceramic-based sealers (BCS) are the different chemical categories used to classify sealants. Calcium silicate-based, calcium phosphate-based, and calcium silicate and phosphate-based combinations are the three categories of bio-ceramic-based sealants [(1)].

The main advantages of BCS are excellent biocompatibility, seal, antibacterial activity and radiopacity. Excellent biocompatibility properties are due to their analogous composition with hard tissues which lead to a regenerative effect. Furthermore, the creation of a chemical bond with the tooth structure is what allows for an effective seal [(4)].

In the 1990s, retrograde fillings made of bio-ceramic materials were first used in dentistry. Later, root healing cements and root canal sealers also used these materials [(5)]. Calcium silicate based sealers have successful results of root canal treatment. They are categorized into two groups, MTA based and non MTA-BCS [(6)].

Tricalcium oxide, tricalcium silicate, bismuth oxide, tricalcium aluminate, tricalcium oxide, tetracalcium aluminoferrite, and silicate oxide make up the majority of the MTA-BCS formula. The chemical and physical characteristics of MTA are also caused by a few other mineral oxides [(7)]. They are dimensional stable, have excellent sealing ability and are not technique sensitive [(8)].

The MTA-BCS have received scientific interest due to their superior properties in comparison to other sealers. Previous studies showed variations in properties of different commercially available MTA-BCS which might affect quality and durability [(9-11)].
The aim of the current study was to evaluate 3 commercially available MTA-BCS sealers regarding flow, film thickness and setting time.

MATERIALS AND METHODS

The 3 commercially available MTA-BCS used in this study are summarized in Table 1.

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Table (1): The materials used in the study regarding scientific name and Commercial, composition, manufacturer, patch number and description.

Scientific name and Commercial	Composition	Manufacturer	Patch No	Description
MTA based bio-ceramic sealer. MTA fillapex sealer	Salicylate resin, bismuth trioxide, fumed silica, fumed silica titanium dioxide, MTA and base resin	Angelus, Londrina, Brazil	102648	Two components
MTA based bio-ceramic sealer. Ceramoseal HBC sealer	Hydroxyapatite, MTA, silicon dioxides, calcium oxide and aluminum oxide, plus calcium silicate, titanium dioxide, barium sulfate and excipients	DM Trust, Egypt	45DK	Single component
MTA based bio-ceramic sealer. Endoseal MTA	Calcium silicates, calcium aluminates, calcium aluminoferrite, calcium sulfates, radiopacifier, thickening agents.	Maruchi; Wonju, Korea	CI210303B	Single component

Sample size calculation and study design:

Sample size was calculated by G power software (G power 3.1.9.7 for windows, heinrich-heine university, Düsseldorf, Germany) using ANOVA (F test): Fixed effects, omnibus, one-way regarding the 3 groups. According to **Abdelrahman *et al.*** [(12)], the effect size was determined (F=0.95) as a minimum of 15 samples (n=5) for each group with an 80% power and a significance threshold of 0.05 for each test were found to be adequate.

A total number of 60 samples were prepared from 3 commercially available MTA-BCS. Samples were divided into 3 groups 20 for each (n =5) per test.

The groups were as follow:

- **Group I:** MTA fillapix sealer.
- **Group II:** Ceramoseal HBC sealer.
- **Group III:** Endoseal MTA sealer.

Flow test: Test was done regarding ISO 6876. A total of 15 samples (n=5) were tested. A 0.05±0.005 ml of sealer in each group was placed on a glass slap with dimensions of 40mm (height) x 40mm (width) x 5mm (thickness) using plastic graduated 100g of load was centrally applied to the slap for 10 minutes after 180 seconds of post-mixing. After that, a digital calliper with 0.01mm precision was used to measure the sealer disk's maximum and minimum diameters. The sample was changed if the difference between the two diameters was more than 1mm.

Film thickness:

Test was done regarding ISO 6876. A total of 15 samples (n=5) were tested. Two 5mm±0.05 thick glass slaps were used. The thickness of the slaps was measured three times by using a digital caliper and considered as initial measurement. A 0.05 ± 0.005ml of the sealers in each group was placed on the center of a glass slap using a plastic graduated insulin syringe. Thereafter, the other slap was positioned carefully over the sealer. A load of 15 kg was placed centrally and vertically on top of the slaps after 180 seconds of post-mixing. Ten minutes after mixing, the load was removed, and a digital caliper was used to take three separate measurements of the combined thickness of the two slaps and the sealer film. The film thickness of the materials was determined from the difference between the means of the two measurements.

Initial and final setting time: Test was done regarding ISO 6876. For initial setting time, a total of 15 samples (n=5) were tested. Samples were prepared by using split metallic molds of 10mm diameter and 2mm height. Wet tissue was used to cover glass slap to provide moisture needed for setting [(9)] then the mold was placed over it. The sealers were applied into the molds according to the manufacturers' instructions for each group at room temperature. The Gilmore device with a freely moved 100g±0.05 weight fixed to, 2mm blunt tip indenter was used to measure initial setting time. After the application of the sealer to the molds the stopwatch was started. The first indentation was done after the end of working time estimated by manufacturer. The indenter was placed perpendicular to the sealer surface for 5sec, repeated every 60 sec till the needle did not leave a discernible indentation. The stopwatch was stopped. The first setup time was established at this moment. The needle tip was carefully cleaned between indentations. The indentations were done 1mm away from the borders and 1mm between each other. For final setting time, another 15 samples (n=5) were tested using Gilmore device with a freely moved 456.5g±0.05 weight fixed to 1.0mm blunt tip indenter.

The same procedures were repeated as done in measuring initial setting time. The final setting time was recorded from the end of mixing till failed to make visible indentation. For both initial and final setting time test, if the full surface of the sample was filled with indentations, the sample was replaced by another one.

Ethical approval:

This study was ethically approved by the Institutional Review Board of the Faculty of Medicine, Minia University. Written informed consent was obtained from all participants. This study was executed according to the code of ethics of the World Medical Association (Declaration of Helsinki) for studies on humans.

Statistical analysis:

The collected data were introduced and statistically analyzed by utilizing the Statistical Package for Social Sciences (SPSS) version 20 for windows. Using the Shapiro-Wilk and Kolmogorov-Smirnov tests, the normality of the data was examined. The mean and standard deviation (SD) were used to summarise the normal data. ANOVA test, followed by Tukey post hoc test, or independent t-test (for final setup time), was used to compare groups. P-values are always two-sided. P value ≤0.05 was considered to be statistically significant.

RESULTS

Regarding flow test, the highest mean value was recorded for Group I (26.5mm) followed by Group III (25.56mm); while the lowest value was recorded for Group II (22.59mm). The mean values of Groups I and III were statistically significant comparing to Group II (Table 2 and Figure 1).

Table (2): Means and Sd for flow test in (mm) in different groups.

Groups	Mean	SD	P-value
Group I (MTA FILLAPEX)	26.50	0.50	0.000*
Group II (CERAMOSEAL HBC)	22.59	1.94	
Group III (ENDOSEAL MTA)	25.56	0.13	

*Significant.

Post-hoc test: There is no statistically significant difference between means with the same superscript letter.

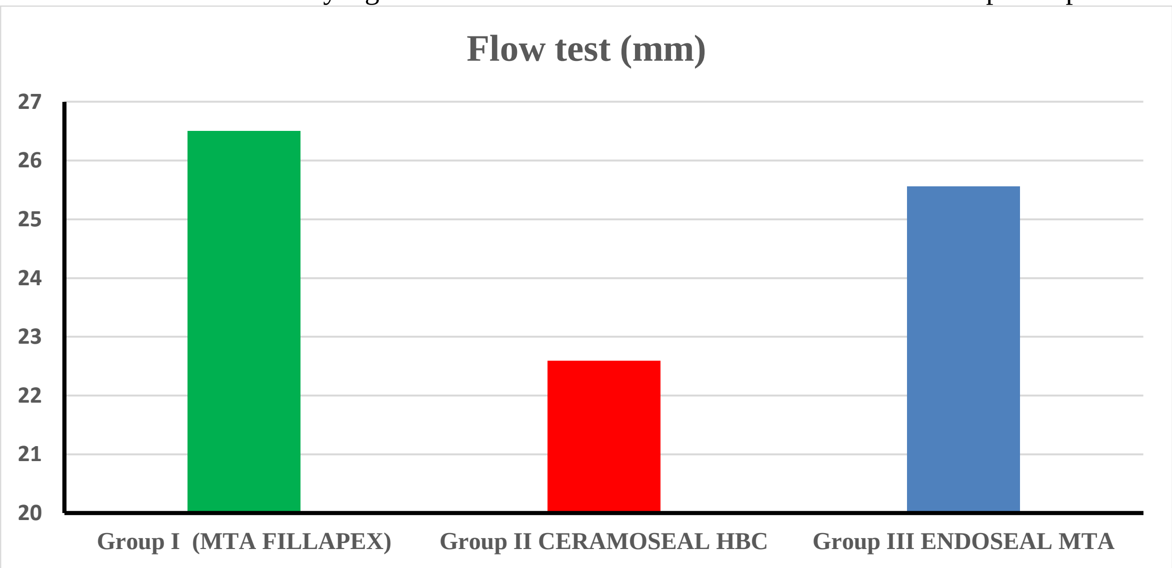

Figure (1): Bar chart illustrating mean values of flow (mm) for different groups.

Regarding film thickness, the highest statistically significant mean value was recorded for Group II (29.4µm), followed by Group I (23µm), while the lowest value was recorded in Group III (15µm). The mean value of Group II was statistically significant comparing to Group III. (Table 3 and Figure 2).

Table (3): Mean and SD for film thickness test (µm) in different groups.

Groups	Mean	Sd	P-value
Group I (MTA FILLAPEX)	23	6.71	0.003*
Group II (CERAMOSEAL HBC)	29.4	4.72	
Group III (ENDOSEAL MTA)	15	3.53	

*Significant.

Post-hoc test: There is no statistically significant difference between means with the same superscript letter.

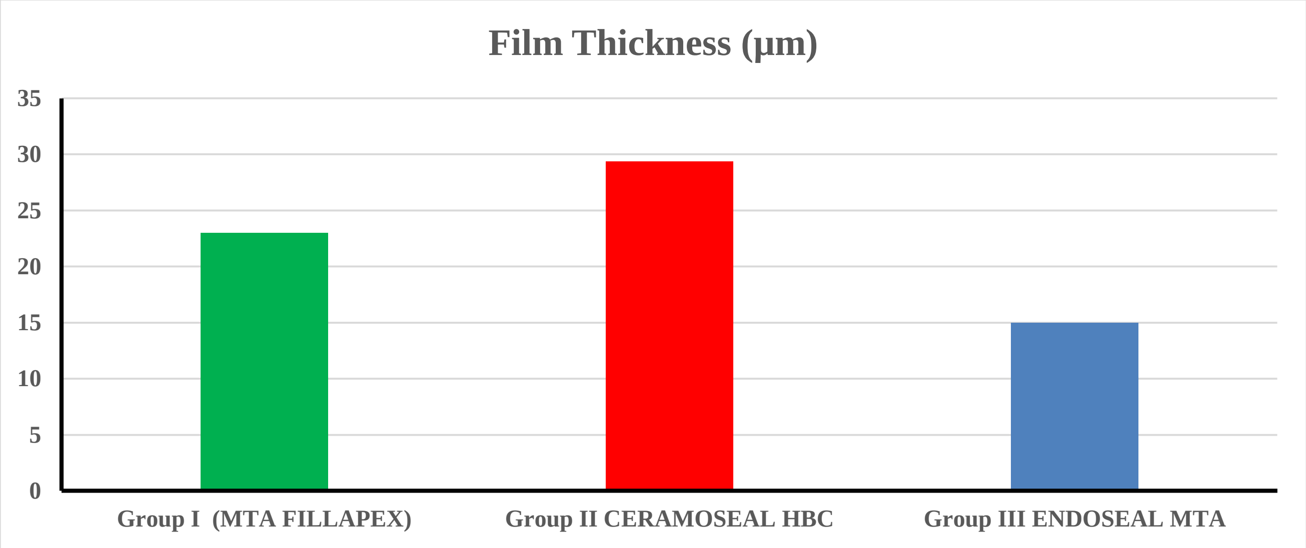

Figure (2): Bar chart illustrating mean values of film thickness (µm) for different groups.

Regarding initial setting time test, the highest statistically significant mean value was recorded for Group I (858.51min) followed by Group II (169.2min), while the lowest value was recorded for Group III (35.2min) (Table 4 and Figure 3).

Table (4): Mean and Sd for initial setting time (min) in different groups.

Groups	Mean	SD	P-value
Group I (MTA FILLAPEX)	858.6	11.82	0.000*
Group II (CERAMOSEAL HBC)	169.2	9.52	
Group III (ENDOSEAL MTA)	35.2	2.86	

*Significant.

Post-hoc test: There is no statistically significant difference between means with the same superscript letter.

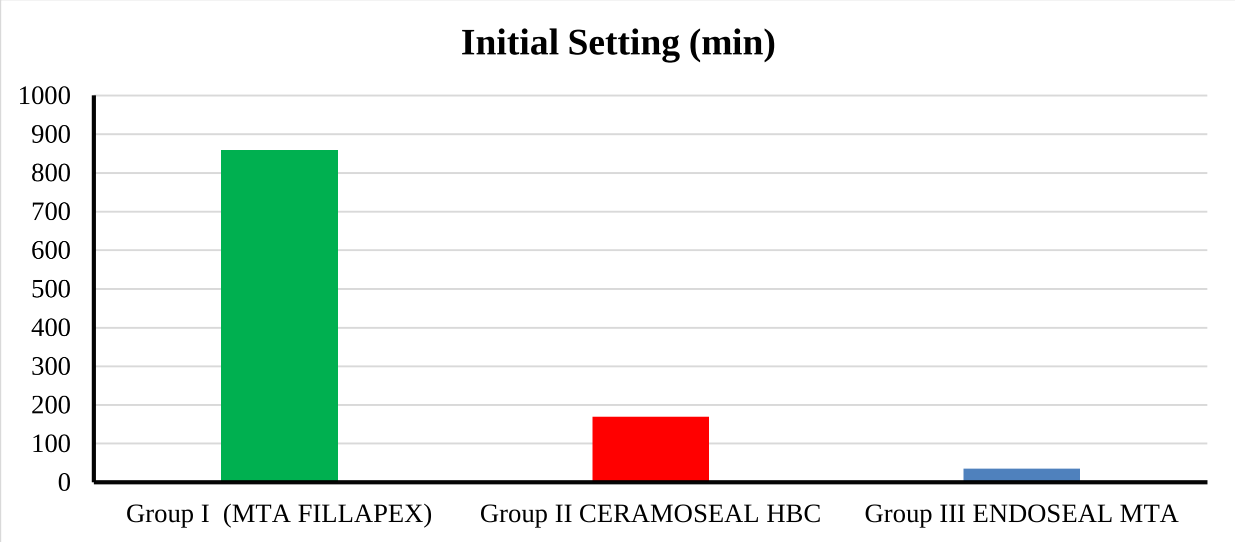

Figure (3): Bar chart illustrating mean values of initial setting time (min) for different groups.

Regarding final setting time test, Group I was unable to set up to one month, Group II recorded a statistically significant higher value (832min), in comparison to the lowest value recorded in Group III (150.4min) (Table 5 and Figure 4).

Table (5): Mean and SD for final setting time (min) in different groups.

Groups	Mean	Sd	P-value
Group I (MTA FILLAPEX)	NA		
Group II (CERAMOSEAL HBC)	832	6.78	0.000*
Group III (ENDOSEAL MTA)	150.4	3.44	

*Significant.

Post-hoc test: There is no statistically significant difference between means with the same superscript letter.

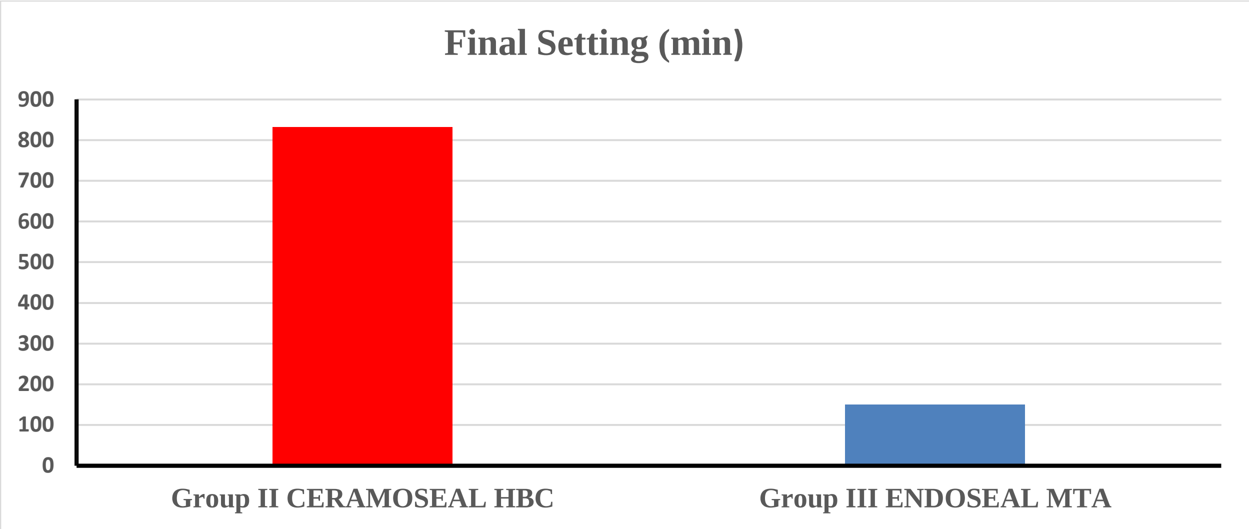

Figure (4): Bar chart illustrating mean values of final setting time (min) for different groups.

DISCUSSION

The MTA-BCS has become quite well known for its top-notch amenities. The quality of the obturation procedure is influenced by the composition and handling characteristics of the root canal sealers. Flow, film thickness and setting time are the most important handling properties as they affected the ability of the sealers to enter narrow and irregular spaces **[13]**.

The high flow of endodontic sealers can affect how the sealers will obturate the gaps between the master and auxiliary cones as well as the accessory canals. Furthermore, sealer thickness showed the tendency of sealers to adapt dimensional change and dissolve over time **[14]**.

Anatomical abnormalities, the isthmus, and lateral canals may be difficult to fill with a sealer with poor flow and a thick layer **[13]**.

For this reason, high flow and low film thickness of sealers are preferred as they could enhance the sealing ability and adaptability of sealers. However, extremely high flow and very low film thickness might lead to the protrusion of sealer across the apical foramen **[15]**.

In light of this, endodontic sealers should fulfil the requirements of ISO no 6786 specifications which stated that, the minimum flow diameter for a root canal sealer is 17mm, and film thickness not more than 50µm **[16]**. Significant elements that can impact the flow and film thickness properties of sealers include the synthesis, filler size, shear rate, temperature, filling technique, and working duration **[17]**.

In this study, all tested sealers showed flow diameter higher than 22mm which was higher than ISO no 6786 limit. The highest mean value was recorded for group I followed by group III, while the lowest statistically significant value was recorded for group II. The high flow value of group I and group III could be attributed to the high amount of resin content in group I and small particle size for both groups **[7,17-19]**. Furthermore, the low flow of group II might be attributed to its composition, particle size and viscosity **[9,20]**.

The lower insignificant mean value of flow obtained for group III sealer compared to group I was in agreement with previous studies **[1,6]**. On the other hand, lower flow value of group I observed in **Zeid *et al.*** **[9]** study might be attributed to the difference at temperature used in both studies.

Regarding to the film thickness for the present study, all tested sealer recorded mean value less than 30µm which is lower than ISO no 6786 limit. Group II had the greatest mean value, followed by Group I, while Group III had the lowest mean value. The high film thickness mean value of group II might be attributed to the same case of low flow that mentioned before **[9,20]**.

Furthermore, the statistically significant low film thickness mean value recorded for group III is in accordance with **Soni *et al.*** **[21]** and **Jo *et al.*** **[22]** and could be attributed to its small particles size **[21,23]**.

On the other hand, the discrepancy between this study and **Zeid *et al.*** **[9]** and **Yamauchi *et al.*** **[24]** studies for group I and III might be attributed to difference of the samples volume and temperature of the glass slaps.

Setting time of the sealer is an important property. Setting time of the sealer should be neither too slow nor too fast to enable the proper obturation of the root canals. Sealers that are unset or partially set can exhibit rapid penetration of bacteria and bacterial by products through the canal. In addition, they may permit dislodgment of free chemicals from the apex that probably be irritant or cytotoxic to the preapical tissues **[1,25]**.

Nevertheless, ISO no 6786 does not state specific range of time for sealers initial and final setting. It is only announced that the sealers which declared by the manufacturer that is set up to 30 min the setting time should not exceed 10% longer than this declaration. Moreover, ISO no 6786 also announced that for sealers which set in time more than 30min up to 4320 min (72hr) or sealers which declared by their manufacturer that they have specific setting time range; the setting time of them should fulfil the manufacturer declaration.

In the present study, all tested groups do not meet the manufacturer declaration except initial setting time for group II. Group II recorded 169.2 (SD 9.52) minutes (2.82 hours), 832 (SD 6.78) minutes (13.87 hours) for initial and final setting time respectively while the manufacture declared that its setting time does not exceed 5 hr. On the other hand, group I recorded 858.6 (SD 11.82) minutes (14.31 hours) for initial setting time and was unable to set up to one month, however the manufacturer declared that it is set at 120 (SD 10) minutes (3 hours). Moreover, group III recorded 35.2 (SD 2.86) minutes, 150.4 minutes (2.5hr) for initial and final setting time respectively while the manufacturer declared that it is set at 13 minutes (SD 10). As known setting of MTA based sealer depend on hydration process [(6)].

The extended setting time of all groups might be due to low amount of moisture that is provided by the wet tissue used during testing the setting time.

The statistically significant extended initial setting time and the inability to achieve final setting time of group I came in agreement with previous studies [(1,9,26)]. On the other hand, **Xuereb *et al.*** [(27)] and **Jafari and Jafari** [(6)] studies showed shorter setting time. The discrepancy could be attributed to difference in testing procedures as they allow the samples to set in contact with the physiologic solution for 14 days.

Furthermore, statistically significant mean value of group III encounter previous studies [(1,28)] and contradict with **Koo *et al.*** [(29)] study which revealed shorter setting time. This attributed to using gypsum mold which provided superior moisture than the wet tissue. Increasing moisture could accelerate the setting reaction [(30)].

CONCLUSION

Within the limitation of this study, it can be concluded that all the tested sealers fulfilled the requirements of ISO specifications no 6876 for flow and film thickness. Regarding setting time, only Group II fulfilled their manufacturer declaration for initial setting as stated by ISO.

- **Conflict of interest:** The investigators declare no conflict of interest.
- **Sources of funding:** The current study didn't receive any specialized grant from funding agencies**.**

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