



Material for evaluation: MTA Fillapex
Tests performed according to International Standard (ISO) 6876

Physical and Mechanical Properties

1 Flow

1.1 Procedure

The two pastes of the root canal sealer were measured in equal parts and mixed until complete homogenization (± 10 s). A volume of 0.05 ml of the mixture was dispensed on the center of a glass slab, which was covered by another glass slab with a 100 g weight on it. This assembly was left alone for 10 minutes.

1.2 Results

Table 1. Flow obtained for each sample and their averages

Samples	Ø 1 (mm)	Ø 2 (mm)	Ø 3 (mm)	Ø Average (mm)
1	28	27	25	26.66
2	28	26	29	27.66
3	28	27	26	27.00

Ø: diameter mm: millimeter

► Conclusion

The material fulfilled all ISO 6876 requirements. It presented flow values that were superior to the minimum criteria required by the norm.

2 Working time

2.1 Procedure

The two pastes of the root canal sealer were measured in equal parts and mixed for different times (10 s, 10 min, 20 min, 30 min, 40 min and 50 min), according to the tables below. A volume of 0.05 ml of the mixture was dispensed on the center of a glass slab, which was covered by another glass slab with a 100 g weight on it. This assembly was left alone for 10 minutes.

The flow was measured with a digital caliper. When the diameter of the sample was 10% less than the initial diameter (mixing time: 10 s), the working time was established.

2.2 Results

Table 1 Flow obtained after different mixing times and their averages between larger and smaller diameters.

Mixing times	Ø1 (mm)	Ø2 (mm)	Ø3 (mm)	Ø4 (mm)	Ø Average
10 s	28.41	28.36	26.67	27.39	27.54
10 min	26.34	26.08	25.92	26.27	26.13
20 min	26.32	25.87	25.85	26.31	26.85
30 min	25.99	26.78	26.01	26.21	26.38
40 min	20.72	22.02	21.61	20.58	21.30
50 min	16.61	16.82	15.98	16.09	16.40

Ø: diameter mm: millimeter s: second min: minute

Table 2 Flow obtained after different mixing times and their averages between larger and smaller diameters.

Mixing times	Ø1 (mm)	Ø2 (mm)	Ø3 (mm)	Ø4 (mm)	Ø Average
10 s	25.64	26.75	25.55	27.63	26.59
10 min	24.64	24.20	24.36	24.60	24.42
20 min	24.56	25.62	25.56	24.50	25.06
30 min	25.80	24.58	25.06	24.84	25.32
40 min	21.95	21.27	21.25	21.45	21.60
50 min	21.05	21.99	21.33	21.44	21.52

Ø: diameter mm: millimeter s: second min: minute

Table 3 Flow obtained after different mixing times and their averages between larger and smaller diameters.

Mixing times	Ø1 (mm)	Ø2 (mm)	Ø3 (mm)	Ø4 (mm)	Ø Average
10 s	25.66	25.61	25.81	25.81	25.65
10 min	23.74	24.61	24.98	24.61	24.36
20 min	23.66	24.30	23.97	24.55	24.10
30 min	24.11	23.89	23.64	23.66	23.87
40 min	24.00	24.77	23.88	23.47	24.12
50 min	23.66	22.44	22.09	22.58	22.87

Ø: diameter mm: millimeter s: second min: minute

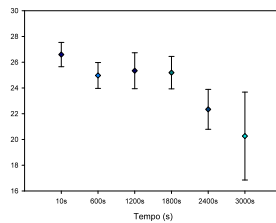


Figure 1. Flow diameter averages and their standard deviations in relation to the different working times.

*Diâmetro de Escoamento means Flow Diameter (mm)

**Tempo means Time (s)

► Conclusion

The material fulfilled all ISO 6876 requirements. It presented an approximate working time of 35 minutes.

3 Film thickness

3.1 Procedure

Two combined glass slabs were measured with a Mitutoyo digital micrometer. Then, an amount of the mixed sealer was dispensed on the center of one of the glass slabs, which was covered by the other glass slab plus a 150 N (15 kg) weight. The sealer filled completely the space between the two glass slabs. After 10 minutes, the thickness of both glass slabs with the sealer between them was measured with the same micrometer.

3.2 Results

Table 1. Film thickness values and averages

Measurements	1 (µm)	2 (µm)	3 (µm)
Glass slabs thickness	9,380	9,380	9,273
Glass slabs + sealer thickness	9,422	9,422	9,314
Sealer film thickness	36	42	41

Film thickness average: $\frac{36+42+41}{3} = 39.6 \mu\text{m}$

Film thickness standard deviation: $3.2 \mu\text{m}$

► Conclusion

Film thickness was not larger than 50 µm according to the ISO tests. The sealer was approved and presented values well within the required limits with a safety margin.

4 Setting time

4.1 Procedure

4.1.1 Gypsum molds were prepared and taken to a dry heat furnace at 37°C and 95% humidity for 24 hours. The test samples were made by filling these molds with the mixed sealer.

4.1.2 Forty (40) minutes after the filling, the first indentation was performed, the tip was cleaned and the test was repeatedly performed in 10 minute intervals, until a deformation or surface alteration would be no longer be visualized.

4.2 Results

The tests were performed in triplicate, under controlled conditions of temperature (25°C) and humidity (< 60%), with the results expressed in minutes.

Table 1. Evaluation of setting times after the indentation tests

Sample	Block 1	Block 2	Block 3
Time (min)	120	130	140

Setting time average: 130 minutes

Standard deviation: 10 minutes

► Conclusion

The root canal sealer presented an average setting time of 130 minutes (2 hours and 10 minutes) with a ± 10 minute variation. ISO does not establish a specific time for materials with setting times longer than 30 minutes. However, such information must be informed by the manufacturer, which was described above.

5 Radiopacity

5.1 Procedure

The sealer was dispensed in the mold, covered by a glass lid and left alone for 2 hours. The sample was placed on a radiographic film beside the 0.5/2.5 area of an aluminum scale. The radiograph was taken.

5.2 Result

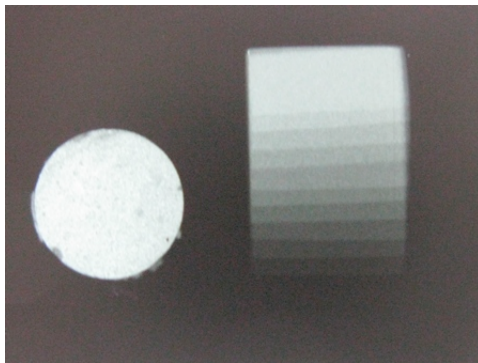


Figure 1. Radiograph of sample and aluminum scale

5.3 Considerations about result

The optical density of the sample must be equal or superior to the area of the aluminum scale that corresponds to a thickness of 3 mm.

Software Image J was used to calculate the optical density in pixels. The material presented a value that was 77% superior to the 3 mm of the aluminum scale.

► Conclusion

The material presented an optical density value that was 77% superior to the 3 mm of the aluminum scale.

6.Solubility

6.1 Results

The solubility test was performed in 3 samples.

Table 1 shows data prior to test and table 2 shows data after test.

Table 1 Initial weight of samples and Petri dish

Material	Weight (g)
Sample 1	1,02708
Sample 2	1,13590
Sample 3	1,18978
Petri dish	46,85867

Table 2. Final weight of samples and Petri dish after solubility challenge

Material	Weight (g)
Sample 1	1,02078
Sample 2	1,13698
Sample 3	1,18296
Petri dish	46,90603

Final variation after solubility test was 0.1%.

Following ISO, after the solubility test, the difference between the initial and final weights determines the amount of material that was solubilized. Such values must be near 0.1% and must not exceed 3%.

► Conclusion

The sealer presented a variation of 0.1% after the solubility test. This value was inferior to 3%, which is the maximum accepted by ISO.