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Biodentine management and setting time with Vicat and Vickers evaluation; a survey-based study on clinicians' experience

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ABSTRACT

Purpose: of this paper was to analyse clinicians' views on the management and handling procedures of the Biodentine tricalcium silicate cement with the following evaluation of the real setting time of the material with two independent physical tests.

Design/methodology/approach: A survey study included 174 clinicians who answered the questionnaire designed to collect opinions on the Biodentine management during endodontic procedures. To verify the setting time of the cement, two independent hardness tests were performed. Macroscopic evaluation was carried out using the Vicat device. Microscopic assessment with subsequent SEM observation was conducted with the aid of the Clemex appliance.

Findings: 43% of respondents using Biodentine in their practice described the setting time as long or definitively too long. One fifth of the dentists surveyed continue dental procedures without waiting. The setting time tests confirmed the existence of two phases of the Biodentine setting process, which corresponds to the general definition of cement setting. After mixing of the material, the initial setting stage lasts for 15 minutes. The next one, described by the authors as "maturation" of Biodentine lasts for 120 minutes.

Research limitations/implications: The material initially sets within 15 minutes, however it is not the end of the process. In certain endodontic procedures the awareness of a longer setting time of Biodentine is essential for decision-making in root canal therapy.

Practical implications: It is advisable to divide the endodontic treatment with Biodentine into two separate appointments.

Originality/value: From the clinicians' perspective the setting time and correct handling of Biodentine are crucial factors in the successful endodontic therapy. The information regarding proper material management is included in this paper.

Keywords: Materials, Biomaterials, Biodentine, Tricalcium silicate cement, Setting time

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BIOMEDICAL AND DENTAL MATERIALS AND ENGINEERING

1. Introduction

Over the past two decades a group of materials named bioceramics has been a subject of an increased interest in dentistry. They are used for restoring and covering the dental hard tissues defects, both in crown and roots of the tooth, because of their biocompatibility, good sealing ability and beneficial biochemical interaction with the local environment [1].

Biodentine (Septodont, Saint-Maur-des-Fosées, France), a self-setting hydraulic calcium-silicate-based cement specifically designed as a "dentin replacement" material became commercially available in 2009 [2]. It is applicable in numerous clinical situations including pulp capping [3-5], dentin replacement [6] and pulpotomy procedures [7,8], the treatment of an immature or open apex [8,9], a root-end retrograde surgical filling [9,10] and perforation repair [9,11,12].

The bioactivity of Biodentine comes from tricalcium and dicalcium silicate phase [13] that constitutes its major component. Along with calcium carbonate, zirconium dioxide and iron oxide it is a part of an encapsulated powder. To activate the setting reaction, the powder is mixed with some liquid containing calcium chloride, a water reducing agent and water [14,15]. The hydration reaction leads to the formation of calcium silicate hydrate (CSH gel) and calcium hydroxide ($Ca(OH)_2$) [3, 14-17]. The total hydration reaction is represented by the following formulas [14]:

$$2(3CaO{\cdot}SiO_2) + 6H_2O \rightarrow 3CaO{\cdot}2SiO_2{\cdot}3H_2O + 3Ca(OH)_2 \quad (1)$$

$$2(2CaO{\cdot}SiO_2) + 4H_2O \rightarrow 3CaO{\cdot}2SiO_2{\cdot}3H_2O + Ca(OH)_2 \quad (2)$$

Calcium hydroxide, one of the products of Biodentine hydration, reacts with the phosphate ions from body fluids and induces hydroxyapatite (HAP) precipitation with water as a by-product. The reaction is summarized below [18]:

$$7Ca(OH)_2 + 3Ca(H_2PO_4)_2 \rightarrow Ca_{10}(PO_4)_6(OH)_2 + 12H_2O \quad (3)$$

Newly formed HAP contains a bone repair and reconstruction material – a non-toxic, active product of Biodentine setting reaction [18]. As shown above, water continues to react with calcium silicates.

The chemical composition of Biodentine and properties of its constituents are described in Table 1.

Table 1.
Biodentine chemical composition and properties of different components [1,11,14,15,19]

Powder	
Tricalcium silicate ($3CaO{\cdot}SiO_2$)	main core material, setting reaction regulator
Dicalcium silicate ($2CaO{\cdot}SiO_2$)	second main core material
Calcium carbonate ($CaCO_3$)	reactive filler (able to react with $CaCl_2$), nucleating agent
Calcium Oxide (CaO)	reactive filler (able to react with $CaCl_2$)
Zirconium dioxide (ZrO_2)	radioopacifier
Iron oxide	pigment, coloring agent
Liquid	
Calcium chloride ($CaCl_2{\cdot}2H_2O$)	setting reaction accelerator
Water reducing agent (hydrosoluble polymer)	water requirement redactor → viscosity decrease, cement handling improvement
Water	solvent

According to the manufacturer, Biodentine was developed as fast-setting cement with the setting time of 12 minutes. It is a significant improvement in comparison with other bioactive cements [15,20]. It was achieved by combining three effects:

1. increasing the particle size (the higher the specific surface, the shorter the setting),
2. adding calcium chloride (the accelerator) to the liquid component,
3. decreasing the liquid intake in the system with the use of hydrosoluble polymer.

Nevertheless, there have been reports providing different data about the setting time ranging from 6.5 minutes to 14 days [21-23].

The material management is user-friendly. A predosed capsule contains powder which is mixed with the liquid by a triturator for 30 seconds at a min. of 4000 rpm. The preparation method and proportions determined by the manufacturer should be respected and applied. They influence the material setting and mechanical properties [11]. Therefore, the outcome of the endodontic treatment is largely dependent on proper cement management. The creamy, putty-like consistency of unset Biodentine makes it easy to handle as it is similar to that of phosphate cement [11,24]. According to comparative studies the Biodentine manipulation comfort is superior to that of MTA [25].

Once Biodentine is applied, it needs time to fully bind, to "mature" so that it can withstand the forces deriving from treatment continuation or mastication. Therefore, from the clinicians' perspective the setting time and correct handling of Biodentine are a crucial factor in successful endodontic therapy.

The aim of this article was to analyse clinicians' opinions on Biodentine management during their daily practice. On the basis of the obtained data the following objective was to evaluate the setting time of Biodentine cement using two complementary methods of hardness testing: Vicat and Vickers tests.

2. Materials and methods

2.1. Survey

The study was conducted among a randomized group of participants of one of the annual Polish dental meetings. The data concerning professional background as well as opinions on various aspects of Biodentine tricalcium silicate cement handling were collected in a specially designed anonymous questionnaire. Except for questions regarding respondents' professional status the issues such as the frequency of Biodentine usage (How often do you use Biodentine?) as well as its clinical applications were raised (In what clinical situations do you use Biodentine? How do you rate Biodentine application comfort on a school scale from 1 to 5 and why?). The questions also concerned clinical protocol involving tricalcium silicate cement placement (For how long do you wait before you continue the treatment while performing Biodentine-related procedures?) and setting time assessment (How would you rate the Biodentine setting time? Why?). The results of the survey inspired the authors to conduct further research evaluating Biodentine setting time.

All collected questionnaires were checked for inaccuracies such as questions left unanswered. All the incomplete forms were excluded from further analysis. Finally, 174 properly answered questionnaires were interpreted and statistically analysed (MS Excel, Office 365, Microsoft, Redmond, Washington, USA).

2.2. Setting time evaluation

Samples preparation

The material used for the research was a commercial Biodentine product – a self-setting hydraulic tricalcium silicate-based cement (Septodont, Saint-Maur-des-Fosées, France). Six samples, three for each testing method, were prepared according to the manufacturer's instructions in terms of proportion and mixing method: 5 drops of dedicated liquid was vertically added to the capsule containing powder. The capsule was closed and mixed for 30 seconds in a mixing device (Dental Mixer SYG200 Amalgamator, Septodont, Saint-Maur-des-Fosées, France). Mixed cement was then transferred with a plastic spatula (part of Biodentine set) to aluminium ring forms placed on a smooth glass non-absorptive plate. The forms were necessary for the cement to keep its shape while being tested. The size of the specimens was equal to the internal size of the form (2 mm high and 10 mm wide). The upper surface was immediately flattened by a second glass plate. The samples were subsequently subjected to setting time and microhardness test with the aid of Vicat device (macroscale) and Clemex device (microscale).

Vicat test

Manual Vicat device (norm PN-EN196-3 [26]) was used to perform the test. Biodentine hardness was assessed in room temperature by lowering the cylindrical flat-ended needle of 1.1 mm in diameter with 300 ± 1 g load onto sample's surface for 5 seconds. The action was repeated every minute from the 2nd minute after material mixing until

the 25th minute and every 5 minutes from the 25th to 145th minute. The depth of the needle penetration was measured with 0.25 mm accuracy.

The time when the needle penetration depth reached 0.25 mm was considered to be the initial setting time, although the test was performed until no visible imprint was marked on the cement surface. The value of 0.1 mm was marked when the indenter left the trace (depth from 0 mm to 0.25 mm).

Vickers test

The Clemex microhardness tester with a diamond pyramid-shaped indenter was used for testing. The Vickers hardness test method was applied. A diamond indenter in the form of a right pyramid with a square base and the angle of 136 degrees between opposite faces was pressed against the cement with the load of 0.1 N (100 G) for 10 seconds. The imprints were made every 5 minutes from the 40th to the 150th minute from material mixing. The test was delayed due to an inability to apply a diamond indenter against a wet and viscous surface of freshly mixed Biodentine (possibility of the device's damage). The position of the sample was visually and manually controlled under the magnification of 400x.

Subsequently, the sample was gold-sprayed and subjected to observations in the scanning electron microscope (HITACHI S - 3000N, Tokyo, Japan) at 1000x magnification with 15 kV accelerating voltage. This allowed the measurement of the actual width of each imprint. Diagonals were measured in millimetres on a monitor screen and converted (according to the scale) into the real width of the imprint (in micrometres).

The time after which the size of the imprints did not show significant deviations was considered to be the termination of the binding process.

3. Results

3.1. Survey

Respondents' profile and cement usage frequency

The major part of respondents were clinicians with professional 5-15-year experience (43.1%), without any specialization (79%), working in private sector (76.4%).

60.3% of respondents reported the use of Biodentine in their daily practice. The others (who do not use the material) were excluded from further analysis. The answers were evaluated for the frequency of cement application – as shown in Figure 1.

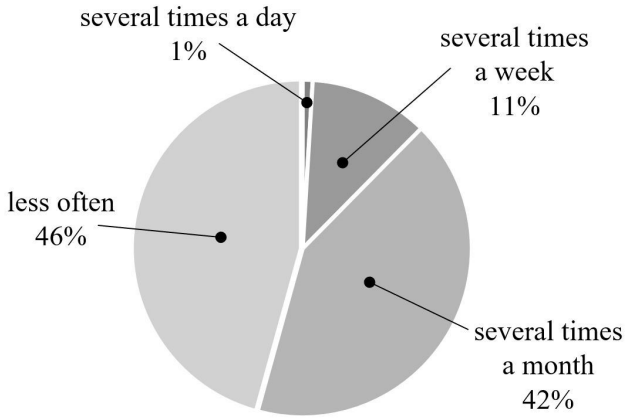

Fig. 1. Frequency of Biodentine usage among respondents

Biodentine applications

According to the survey results, the cement is applied in all of its indication fields. Table 2 shows them in popularity order (more than one answer was allowed). The respondents most commonly use Biodentine while performing indirect pulp capping.

Table 2.
Biodentine applications in popularity order

No.	Biodentine applications [1,2,11,14]	Number of respondents
1.	Indirect pulp capping	85
2.	Direct pulp capping	68
3.	Perforation closure	64
4.	Internal resorptions	30
5.	Pulpotomy	28
6.	Apexification	26
7.	Final dentine reconstruction	18
8.	Surgical canal backfill	15
9.	Root canal filling	12
10.	Deep pericervical and root lesions	10

Material management

The clinicians were asked to rate Biodentine application comfort on a school scale from 1 (not comfortable at all) to 5 (very comfortable). The results are shown in Figure 2.

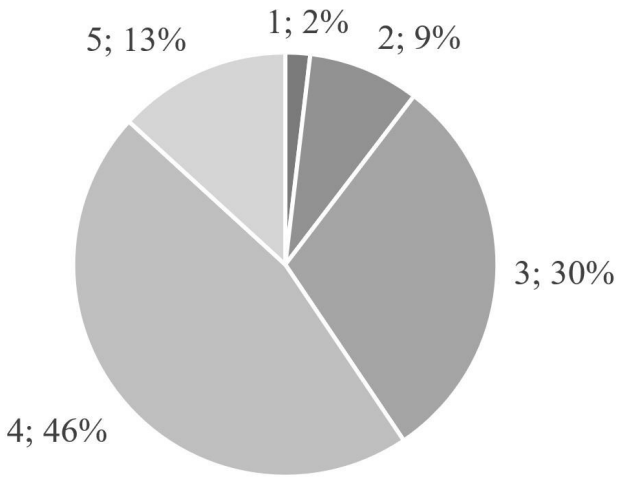

Fig. 2. Material application comfort assessment on scale from 1 (not comfortable at all) to 5 (very comfortable)

Justifications of the clinicians' opinions on Biodentine management are presented below (not all the respondents justified their opinion):

- consistency comfortable in application,
- is more comfortable to use than MTA,
- good consistency and setting time,
- long setting time,
- too much material in a capsule,
- inhomogeneous,
- fluidization of the material during application,
- does not always have the same consistency after mixing,
- long setting time,
- adheres too much to the instruments,
- difficult to put in a canal,
- despite following the instructions for Biodentine preparation, the consistency is not very repeatable and is sometimes too liquid,
- liquid consistency,
- hard to apply, not plastic,
- different consistency each time, after application it often falls off or crumbles,
- when needed, it is needed quickly – I would like to see an "aplicap" mechanism,
- produced in encapsulated form, large material losses e.g. in the case of perforation repair,
- difficult application,
- it crumbles,
- not leak-proof.

The Biodentine setting time assessment consisted of two questions. The first one evaluated the opinion on setting duration: "How would you rate the Biodentine setting time?". The other was meant to determine clinical behavior during procedures involving tricalcium silicate cement: "While performing the most frequent Biodentine-related procedures you have to wait before you continue the treatment. How long?". The results are illustrated on the charts in Figure 3.

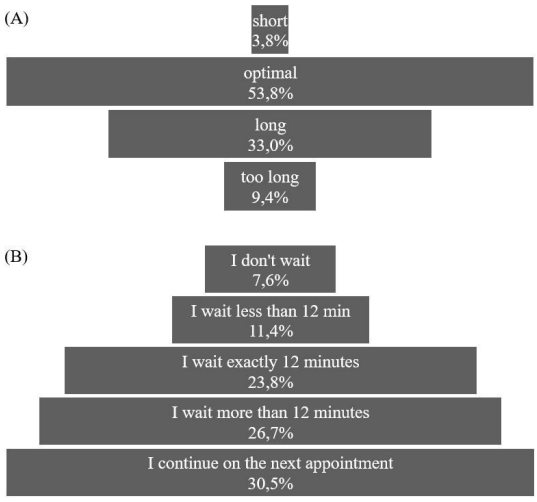

Fig. 3. Biodentine setting time evaluation (A) and clinical compliance (B) – percentage of respondents

The respondents were asked to give explanation for their judgement of the setting time length. As it was not a mandatory question, only a limited number of clinicians taking part in the study replied.

Those who rated it as optimal stated that a perfect setting time should be shorter, but it is true that the problem of long-setting MTA has been eliminated. They also admitted that there is enough time to work with Biodentine, but the treatment time is not too extended, so the patient can easily endure the whole procedure.

The clinicians, for whom the Biodentine setting time was long or too long, complained that the 12 minutes out of 1 hour appointment is definitively too much. They also pointed out that it is not possible to continue dental procedures during the same appointment or that the cement cannot be layered with another material in a very short time, as it is often an unreliable procedure. It was noticed that the time needed to maintain the material shape in conservative procedures does not seem to be adapted to the modern style of work and that some indications make it difficult to keep the treatment field dry, which affects the setting time and hinders their work.

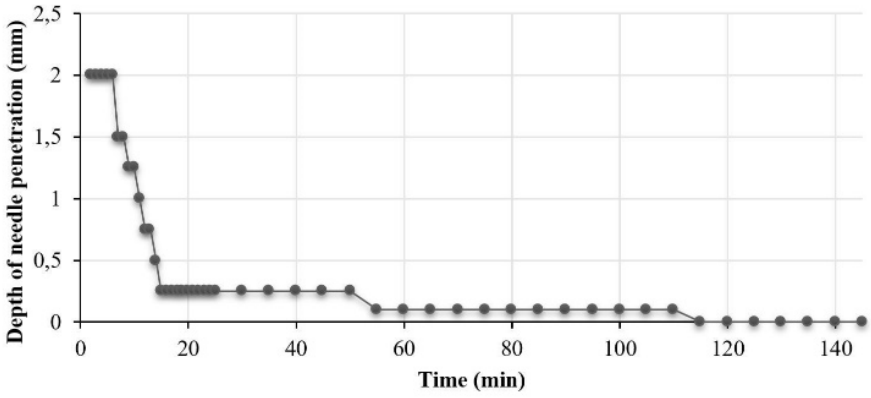

Fig. 4. Depth of Biodentine sample penetration in the Vicat device as a function of time

The last request in the questionnaire regarded some final thoughts on Biodentine hydraulic tricalcium silicate cement. The prevailing commentaries concerned its encapsulated, predosed form as well as long setting time. The respondents expressed their readiness to use capsules with smaller doses of the material, as in many cases they deal with a significant cement waste. Many wished that the time before being able to make final restoration on Biodentine layer was shorter.

3.2. Setting time evaluation

Vicat test

Figure 4 shows a graph illustrating the change in the depth of the Vicat needle penetration into the tested material as a function of time.

The depth of Vicat needle penetration was more and more shallow, from 2 mm in the first 5 minutes after mixing of the material to 0.25 mm in the 15th minute. After that time it stayed the same until the 55th minute. Insignificant needle traces were present up to the 110th minute. Eventually, no visible imprints were left on the cement surface.

Vickers test

The combined images of the Clemex diamond indenter imprints on the surface of Biodentine obtained in a scanning electron microscope are presented in Figure 5.

They do not have the shape expected from the Vickers indenter. They look rather like imprints made with the Knoop penetrator due to the fact that the sample during SEM observation was expressly tilted in order to obtain a clearer image of the imprints.

The real sizes of the imprints are presented in Table 3. The changes in their dimensions are shown as a function of time in Figure 6. After the 120th minute since the material mixing the chart line has visibly flattened, which indicates the stabilization of the system and the end of hardening process.

Table 3.
Clemex indenter imprints' actual size

No.	Time, min	Size, µm
1.	40	392
2.	45	367
3.	50	no data
4.	55	342
5.	60	300
6.	65	283
7.	70	250
8.	75	238
9.	80	196
10.	85	188
11.	90	179
12.	95	171
13.	100	175
14.	105	154
15.	110	150
16.	115	150
17.	120	123
18.	125	117
19.	130	113
20.	135	108
21.	140	117
22.	145	117
23.	150	108

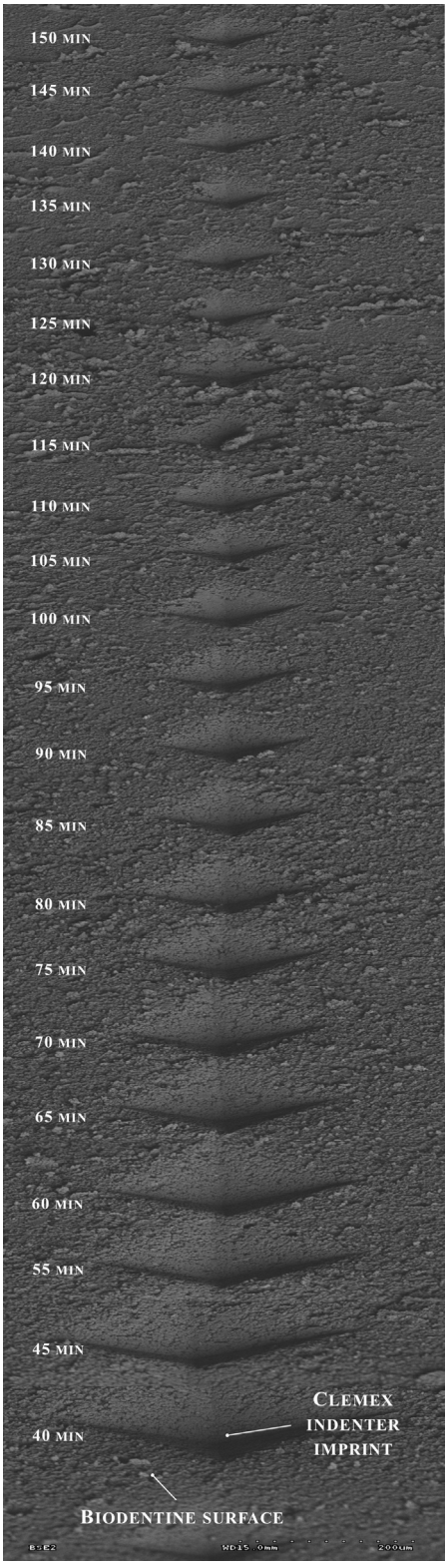

Fig. 5. SEM image of Clemex imprints on the surface of Biodentine

4. Discussion

The Biodentine tricalcium silicate cement is a promising material for covering defects or replacing hard tissues of a tooth. The longer it is available on the professional market, the more clinicians are going to use it, given a wide field of indications for its application. Therefore, the knowledge of proper material management is crucial.

The survey-based study is selective. It always concerns a specific group of respondents that the authors are aiming for. It is worth noticing the fact that in the presented study the popularity of each Biodentine application shows a trend among non-specialists, in majority. The respondents claim indirect pulp capping to be the most common reason for the use of Biodentine. This may be connected with the frequency of such a treatment method among the group of general dentists, who constitute a major part of the surveyed clinicians.

Consequently, quite often negative opinions on Biodentine application comfort arise from the improper cement management (i.e. use of the saline solution instead of a dedicated liquid) or the use of a random triturator. The authors purposely chose a mixing device that was produced by the same company as the material. During sample preparation no deviations from the regular consistency of Biodentine were noticed. The freshly mixed paste was, with no exceptions, homogenous and repetitive.

The discrepancies in the current research concerning the setting time of Biodentine may be difficult to interpret due to different test methods, environments used [1] or differing notions of the setting time.

According to American Society for Testing and Materials (ASTM), the time of setting is defined as the time elapsed from the addition of the fluid to a cementitious powder, until the mixture reaches a specified degree of rigidity measured by a specific procedure. It is a gradual and continuous process [27]. The level of rigidity is determined, based on the penetration resistance and the use of indicated methods, such as the Vicat technique. The norms are dividing the setting process into stages: the time of initial setting and the time of final setting, depending on the resistance to penetration by a probe. The initial setting time is the time when the paste starts losing its plasticity. At the time of final setting, the concrete becomes rigid and it fractures rather than flows as increasing stress is applied. The physical behavior of Biodentine, which derives from Portland cement does not differ in this research from the above description. The available literature on Biodentine

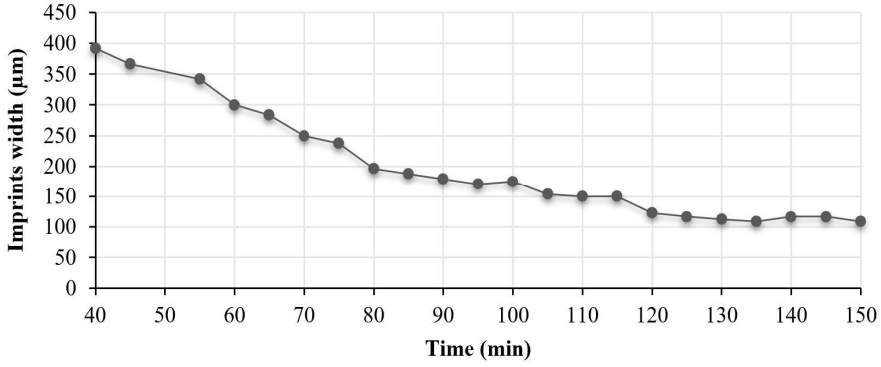

Fig. 6. Changes in imprints width as a function of time

setting time usually does not clearly indicate the stage of setting evaluated. In Biodentine Scientific File the setting time is clearly divided into initial (6 minutes) and final (10.1 minutes) [15]. The result was confirmed using ISO-standardized method with the Gillmore needle – a method alternative to the Vicat technique.

The standards applied refer equally to constructional cements and dental cements. There is a need to ponder on the appropriateness of those standards. In dentistry the requirements of the set cement differ from those used in construction.

For the setting time evaluation the authors chose the Vicat and Vickers methods. While the results are informative and clinically relevant, there is still a need for further or longer-lasting research.

The Vicat method used to evaluate macroscopically the initial hardening time of Biodentine caused some methodological difficulties. The penetration time needed for the needle to reach the depth of 0,5 mm is considered to be the setting time for the constructional cements [28]. For ceramics the depth equals 0,2mm. In the scarce literature on dental biocements Vicat testing it is assumed that this value is 0 mm [29]. In order to average out the above outlines and meet the measurement capabilities of the device used, the boarder value adopted in the research was estimated at 0.25 mm, although the test was performed until the imprint which was marked on the cement surface became invisible.

It must be emphasized that the time of setting measured by this method will not necessarily provide exactly the same results as the setting time of hydraulic cement paste measured by other methods.

The Vickers method was used to microscopically evaluate the hardening process. It was performed after the cement initial hardening phase in order to observe its final transformation due to the viscosity of the freshly mixed paste which could stick to the diamond indenter and falsify the results. The SEM observation had to be modified when compared to the standard technique of Vickers hardness testing. During standard measurements both diagonals of the imprint are measured, the average is calculated and its value is used for hardness HV calculations [30]. The authors adapted a new, easier technique that was adequate for the purpose of this research. The sample observed in SEM was tilted in order to capture a better delimitation of the imprint and a more definitive view of just one diagonal. The real width of the imprint could be still measured. However, in this study the authors did not search for the exact size of the imprint but were rather aiming for observing a trend in changing the dimension. The aim was to determine the time when this change became minimal, which was interpreted as the stabilization of the process.

After combining the results from both the setting tests it can be stated that the dynamics of the process drops drastically after the first quarter but the binding process continues up to 2 hours. Therefore, the Biodentine setting can be divided into two parts: the first – the initial setting and the other – maturation or the final setting (as shown in Fig. 7). This conclusion points to the cementitious nature of Biodentine, as described in ASTM standards. Based on the results of this research, the setting time given by the manufacturer which is 9 to 12 minutes [15] can be related to the first phase of the binding process.

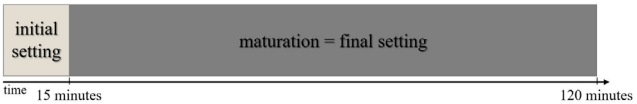

Fig. 7. Biodentine setting divided into two phases

During another research conducted by the authors it was stated that the 12-minute setting time of Biodentine recommended by the manufacturer is too short for the cement to withstand the forces implied during the continuation of the clinical endodontic procedures like cleaning and shaping of the canals and the use of PUI (Passive Ultrasonic Irrigation). It was impossible to mechanically shape/polish the 12-minute Biodentine samples due to the material's instability. Furthermore, when immersed in NaOCl solution with and without ultrasonic activation, they became severely damaged or even totally disintegrated [23].

To explain the two phases of setting reaction it is necessary to understand the physical and chemical level of Biodentine's setting. Mixing powder with liquid triggers the reaction of hydration of tricalcium silicate. It is achieved by gradual dissolution of tricalcium silicate and formation of calcium silicate hydrate. C-S-H gel grows on the unreacted C-S grains and consequently fills in the spaces between them, leading to a decrease in material porosity and increase in its compressive strength [11]. The hardening process results from the formation of crystals that are deposited in a supersaturated solution [31]. The gel layers are relatively impermeable to water, which slows down the further process of hydration of tricalcium silicate [14].

Compared to other bioactive materials, the decrease in the setting time is achieved by increasing the specific surface of tricalcium silicate particles. Also, calcium chloride added to the system acts as an accelerator. Finally, by adding the water reducing agent the manufacturer decreased the liquid content in the system [1,15]. These three features enable the Biodentine cement to set within 9 to 12 minutes (according to the manufacturer).

While the setting time is undoubtedly shorter when compared with the golden-standard MTA [12,25,32] its exact value is arguable.

5. Conclusions

A thorough analysis of the survey indicated that the setting time of the evaluated cement is one of the major factors influencing the clinical protocol. Furthermore, not all the clinicians do respect the material management instructions.

The setting time provided by the manufacturer corresponds with the initial, dynamic phase of the hardening process. Because of the existence of a subsequent maturation phase it is advisable to divide the treatment into two separate visits. The change in clinicians' approach is therefore needed.

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The authors declare that there is no conflict of interests regarding the publication of this paper.

References

[1] A.E. Dawood, P. Parashos, R.H.K. Wong, E.C. Reynolds, D.J. Manton, Calcium silicate-based cements: composition, properties, and clinical applications, Journal of Investigative and Clinical Dentistry 8/2 (2017) e12195. DOI: https://doi.org/10.1111/jicd.12195

[2] Ö. Malkondu, M. Karapinar Kazandağ, E. Kazazoğlu, A review on biodentine, a contemporary dentine replacement and repair material, BioMed Research International 2014 (2014) Article ID 16095. DOI: https://doi.org/10.1155/2014/160951

[3] J. Camilleri, Hydration characteristics of Biodentine and Theracal used as pulp capping materials, Dental Materials 30/7 (2014) 709-715. DOI: https://doi.org/10.1016/j.dental.2014.03.012

[4] C.J. Tomás-Catalá, M. Collado-González, D. García-Bernal, R.E. Oñate-Sánchez, L. Forner, C. Llena, A. Lozano, J.M. Moraleda, F.J. Rodríguez-Lozano, Biocompatibility of New Pulp-capping Materials NeoMTA Plus, MTA Repair HP, and Biodentine on Human Dental Pulp Stem Cells, Journal of Endodontics 44/1 (2018) 126-132. DOI: https://doi.org/10.1016/j.joen.2017.07.017

[5] L. Awawdeh, A. Al-Qudah, H. Hamouri, R.J. Chakra, Outcomes of Vital Pulp Therapy Using Mineral

Trioxide Aggregate or Biodentine: A Prospective Randomized Clinical Trial, Journal of Endodontics 44/1 (2018) 1603-1609. DOI: https://doi.org/10.1016/j.joen.2018.08.004

[6] J. Camilleri, Investigation of Biodentine as dentine replacement material, Journal of Dentistry 41/7 (2013) 600-610. DOI: https://doi.org/10.1016/j.jdent.2013.05.003

[7] C. Cuadros-Fernández, A.I. Lorente Rodríguez, S. Sáez-Martínez, J. García-Binimelis, I. About, M. Mercadé, Short-term treatment outcome of pulpotomies in primary molars using mineral trioxide aggregate and Biodentine: a randomized clinical trial, Clinical Oral Investigations 20 (2016) 1639-1645. DOI: https://doi.org/10.1007/s00784-015-1656-4

[8] N.A. Taha, S.Z. Abdulkhader, Full Pulpotomy with Biodentine in Symptomatic Young Permanent Teeth with Carious Exposure, Journal of Endodontics 44/6 (2018) 932-937. DOI: https://doi.org/10.1016/j.joen.2018.03.003

[9] M. Torabinejad, M. Parirokh, P.M.H. Dummer, Mineral trioxide aggregate and other bioactive endodontic cements: an updated overview – part II: other clinical applications and complications, International Endodontic Journal 51/3 (2018) 284-317. DOI: https://doi.org/10.1111/iej.12843

[10] G. Caron, J. Azérad, M.O. Faure, P. Machtou, Y. Boucher, Use of a new retrograde filling material (Biodentine) for endodontic surgery: Two case reports, International Journal of Oral Science 6 (2014) 250-253. DOI: https://doi.org/10.1038/ijos.2014.25

[11] I. About, Biodentine: from biochemical and bioactive properties to clinical applications, Giornale Italiano Di Endodonzia 30/2 (2016) 81-88. DOI: https://doi.org/10.1016/j.gien.2016.09.002

[12] A.K. Kakani, C. Veeramachaneni, C. Majeti, M. Tummala, L. Khiyani, A review on perforation repair materials, Journal of Clinical and Diagnostic Research 9/9 (2015) ZE09-ZE13. DOI: https://doi.org/10.7860/JCDR/2015/13854.6501

[13] J. Camilleri, L. Grech, K. Galea, D. Keir, M. Fenech, L. Formosa, D. Damidot, B. Mallia, Porosity and root dentine to material interface assessment of calcium silicate-based root-end filling materials, Clinical Oral Investigations 18 (2014) 1437-1446. DOI: https://doi.org/10.1007/s00784-013-1124-y

[14] H. Singh, M. Kaur, S. Markan, P. Kapoor, Biodentine: A Promising Dentin substitute, JBR Journal of Interdisciplinary Medicine and Dental Science 2/5 (2014) 140. DOI: https://doi.org/10.4172/2376-032X.1000140

[15] Septodont, Biodentine Active Biosilicate Technology Scientific File, In Vitro, 2010.

[16] L. Grech, B. Mallia, J. Camilleri, Characterization of set Intermediate Restorative Material, Biodentine, Bioaggregate and a prototype calcium silicate cement for use as root-end filling materials, International Endodontic Journal 46/7 (2013) 632-641. DOI: https://doi.org/10.1111/iej.12039

[17] J. Camilleri, F. Sorrentino, D. Damidot, Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, Biodentine and MTA Angelus, Dental Materials 29/5 (2013) 580-593. DOI: https://doi.org/10.1016/j.dental.2013.03.007

[18] K. Koch, D. Brave, A new day has dawned: the increased use of bioceramics in endodontics, Dentaltown April (2009) 39-43. Available from: https://www.dentaltown.com/Images/Dentaltown/magimages/0409/DTApr09pg39.pdf

[19] R. Chalas, E. Mielko, J. Zubrzycka-Wrobel, J. Nowak, A chemical activity evaluation of two dental calcium silicate-based materials, Current Issues in Pharmacy and Medical Sciences 28/2 (2015) 89-91. DOI: https://doi.org/10.1515/cipms-2015-0051

[20] A.A. Madfa, F.A. Al-Sanabani, N.H. Al-Qudami Al-Kudami, Endodontic Repair Filling Materials: A Review Article, British Journal of Medicine and Medical Research 4/16 (2014) 3059-3079. DOI: https://doi.org/10.9734/BJMMR/2014/9079

[21] L. Grech, B. Mallia, J. Camilleri, Investigation of the physical properties of tricalcium silicate cement-based root-end filling materials, Dental Materials 29/2 (2013) e20-e28. DOI: https://doi.org/10.1016/j.dental.2012.11.007

[22] C. Villat, V.X. Tran, N. Pradelle-Plasse, P. Ponthiaux, F. Wenger, B. Grosgogeat, P. Colon, Impedance methodology: A new way to characterize the setting reaction of dental cements, Dental Materials 26/12 (2010) 1127-1132. DOI: https://doi.org/10.1016/j.dental.2010.07.013

[23] A. Palatyńska-Ulatowska, K. Buła, L. Klimek, Influence of sodium hypochlorite and ultrasounds on surface features and chemical composition of Biodentine tricalcium silicate-based material, Dental Materials Journal (2020) online ahead of print. DOI: https://doi.org/10.4012/dmj.2018-430

[24] S. Priyalakshmi, M. Ranjan, Review on biodentine-A bioactive dentin substitute, IOSR Journal of Dental and Medical Sciences 13/1(ver. III) (2014) 13-17.
[25] M. Kaur, H. Singh, J.S. Dhillon, M. Batra, M. Saini, MTA versus biodentine: Review of literature with a comparative analysis, Journal of Clinical and Diagnostic Research 11/8 (2017) ZG01-ZG05. DOI: https://doi.org/10.7860/JCDR/2017/25840.10374
[26] PN-EN 196-3 – Cement testing methods Determination of setting times and volume stability (in Polish).
[27] ASTM C125-20, Standard Terminology Relating to Concrete and Concrete Aggregates, ASTM International, West Conshohocken, PA, Book of ASTM Standards, 04.02.2020, 1-9.
[28] PN-EN 13279-2 – Gypsum binders and plasters Part 2: Test methods (in Polish).
[29] P. Torkittikul, A. Chaipanich, Investigation of the mechanical and in vitro biological properties of ordinary and white Portland cements, ScienceAsia 35 (2009) 358-364. DOI: http://dx.doi.org/10.2306/scienceasia1513-1874.2009.35.358
[30] B. Bolhari, M.H. Nekoofar, M. Sharifian, S. Ghabrai, N. Meraji, P.M.H. Dummer, Acid and microhardness of mineral trioxide aggregate and mineral trioxide aggregate-like materials, Journal of Endodontics 40/3 (2014) 432-435. DOI: https://doi.org/10.1016/j.joen.2013.10.014
[31] A.J. Allen, J.J. Thomas, H.M. Jennings, Composition and density of nanoscale calcium-silicate-hydrate in cement, Nature Materials 6 (2007) 311-316. DOI: https://doi.org/10.1038/nmat1871
[32] D.P. Parirokh, M. Torabinejad, P.M.H. Dummer, Mineral trioxide aggregate and other bioactive endodontic cements: an updated overview–part I: vital pulp therapy, International Endodontic Journal 51/2 (2018) 177-205. DOI: https://doi.org/10.1111/iej.12841