



# Correlations Composition – Processing – Properties for Zincophosphate Cements

CRISTINA DUMITRESCU<sup>1\*</sup>, ION TEOREANU<sup>2</sup>

<sup>1</sup>S.C CEPROCIM S.A, Bucharest, Romania, 6 Preciziei Blvd, Bucharest, Romania

<sup>2</sup>University Politehnica Bucuresti, 1-7, Polizu Str., Bucharest, Romania

*In the present paper, the correlations composition-processing-reactivity (of solid-basic component with liquid acid) reflected in the characteristic properties of zincophosphate cements (shown in operation and use) were studied. Ten compositions of zincophosphate cements were involved in our research from ten solid components of different compositions and from the same liquid component with adequate concentration. Hardening behaviour of the prepared cements through determinations regarding standard consistency-measure of cement slurries workability, setting time, acidity of cement slurries hardened at different periods of time – by pH measurements (including measurements of pH for cements components), solubility of hardened cements and compressive strength were investigated. Correlations composition-processes-properties were emphasized, establishing the determinant influence factors of zincophosphate cements properties and the mechanisms of reaction and hardening process of these cements were suggested. Zincophosphate cements with superior properties were obtained.*

*Keywords: zincophosphate cement, solid component, liquid component, acid-base reactions, hardening*

Zincophosphate cement is a binder whose hardening is based on oxide-acid reactions, in aqueous environment. Similar to silicate binders, reaction products are hydro-compounds, in this case – hydro-phosphates, that proceed from initial coagulation structures to polycondensation and polycondensation-crystallization structures, the last ones being decisive for structural-mechanical properties of hardened system [1-4]. Polycondensation and polycondensation-crystallization structures formed must be as low tensioned as possible. Consequently, their forming and thus, of oxide-acid reaction products is necessary to have a kinetic adequate to zincophosphate cements specificity, determined by solid-basic component reactivity, in ratio with liquid-acid component [5]. The reactivity of the two zincophosphate cement components, beside their composition and processing parameters when casting the binder (conditions of sintering and grinding finess – for powdery component, neutralization degree and water ratio

for liquid component, respectively) is determinant for the characteristic of the last properties [5, 6].

Present paper deals with the correlations composition-processing-reactivity (of solid-basic component, with liquid-acid), reflected in characteristic properties – in operation and in use of cements. The considered properties are: physico - chemical, and structural-mechanical, respectively.

## Experimental part

In order to conduct the investigations in ZnO-P<sub>2</sub>O<sub>5</sub>-H<sub>2</sub>O system and derivates, materials of high grade were used – Table 1.

The corresponding powdery component, ten precursor mixtures, were studied; their composition is presented in table 2.

The precursor powders were obtained through grinding in agate mills up to a residue of 0% on the sieve 10000

**Table 1**  
PRECURSOR MATERIALS USED AT ZINCOPHOSPHATE CEMENTS PREPARATION

Precursor materials for solid-powder component							Precursor materials for liquid component				
Material denomination	Zinc oxide	Magnesium oxide	Silica oxide	Aluminium oxide	Calcium carbonate	Calcium fluoride	Material denomination	Zinc oxide	Magnesium oxide	Aluminium hydroxide	Orthophosphate acid
Content in useful substance	99.20% ZnO	92.87% MgO	95.42 % SiO <sub>2</sub>	99.9% Al <sub>2</sub> O <sub>3</sub>	99.9% CaCO <sub>3</sub>	99.99% CaF <sub>2</sub>	Content in useful substance	99.20% ZnO	92.87% MgO	99.20% Al(OH) <sub>3</sub>	85-89% H <sub>3</sub> PO <sub>4</sub> ρ=1.75 g/ml

Symbol of precursor mixture	Chemical composition of the precursor mixtures of solid component, %					
	ZnO	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	CaF <sub>2</sub>
a <sub>1</sub>	94.00	6.00	-	-	-	-
a <sub>2</sub>	89.00	10.00	1.00	-	-	-
a <sub>3</sub>	90.20	8.40	1.40	-	-	-
a <sub>4</sub>	89.20	8.40	1.40	-	-	-
a <sub>5</sub>	88.00	8.00	2.00	2.00	-	1.0
a <sub>6</sub>	88.50	8.50	1.00	2.00	-	-
a <sub>7</sub>	88.00	9.00	1.00	2.00	-	-
a <sub>8</sub>	85.00	10.00	4.00	1.00	-	-
a <sub>9</sub>	88.00	8.50	0.50	2.00	1.00	-
a <sub>10</sub>	88.00	8.50	0.50	2.00	1.00	0.06

\* email: cristina.dumitrescu@ceprocim.ro

**Table 2**  
CHEMICAL COMPOSITION OF THE MIXTURES USED FOR POWDERY COMPONENT OF ZINCOPHOSPHATE CEMENTS

**Table 3**  
LIQUID COMPOSITION AT ZINCOPHOSPHATE CEMENTS PREPARATION

Liquid component symbol	Chemical composition (analytical), %				Phase composition (calculated), %		
	H <sub>3</sub> PO <sub>4</sub>	Al <sub>2</sub> O <sub>3</sub>	ZnO	Water	H <sub>3</sub> PO <sub>4</sub> free	H <sub>3</sub> PO <sub>4</sub> combined	Phosphates
L	55.9	3.9	3.2	37.0	38.55	17.35	23.90

**Table 4**  
MAIN CHARACTERISTICS AND PROPERTIES OF ZINCOPHOSPHATE CEMENTS

Cement symbol	Standard consistency, g/0.5 ml	Setting, min.	Compressive strength Rc (24h) - MPa	Film thickness, cement, μm	Solubility in water, mg P <sub>2</sub> O <sub>5</sub> /g	pH
STR 1518/87	1	4 ½ - 8 ½	70	25-40	2.0	-
Z <sub>1</sub>	1.2	3 ½	65	45	1.80	5.10
Z <sub>2</sub>	1.3	6 ¼	90	20	0.15	4.80
Z <sub>3</sub>	1.2	4 ½	75	30	0.50	4.80
Z <sub>4</sub>	1.25	4 ½	80	30	0.50	4.80
Z <sub>5</sub>	1.25	5 ½	70	25	0.50	4.95
Z <sub>6</sub>	1.3	6 ½	98	25	0.15	5.20
Z <sub>7</sub>	1.3	7 ¼	98	15	0.17	5.20
Z <sub>8</sub>	1.2	4	85	35	0.80	5.10
Z <sub>9</sub>	1.35	7	90	20	0.50	5.20
Z <sub>10</sub>	1.35	6 ½	100	15	0.03	5.60

mesh/cm<sup>2</sup> (<60μm). Ground mixtures were thermally treated at temperatures of 1100, 1200, 1300 and 1400°C, with a plateau of 2, 4 and 6 h. The cooling was sudden or slowly, respectively (period of 24 h). The treated (sintered) material was also ground in agate ball mills up to a residue of 0% on the sieve of 6000 mesh/cm<sup>2</sup> (< 40 μm). The liquid composition used at zincophosphate cements preparation is given in table 3. The liquid phase was obtained through partial neutralization of orto-phosphoric acid (concentration 89% and density ρ=1.75 g/mL) with aluminium hydroxide and zinc oxide, at room temperature or on water bath (80-100°C). The adjustment, after case, of the density was made through water proportion variation (using distillate water).

Properties study - in operation and in use of cements was conducted according to case, on pastes of standard consistency that correspond to a certain masic solid/liquid ratio, depending on their reactivity (dependence on composition and processing parameters of the two components). In our determinations the methods forseen in Romanian (SR) and foreign (ISO, BS, DIN, NF) standards or included in ADA/no.8 specification (American Dental Association) [8] were considered. Evaluation conditions of the properties were considered by taking into account behaviour specifications - at casting and in usage, of zincophosphate cements (from physical, chemical and mechanical structural aspect). Cement setting, mechanical strength to be developed, physico-chemical and structural-mechanical stability (workability, solubility, pH) are the most relevant properties values which must be related to compositional and processing parameters, for adjustment of the reactivity of solid-basic component with liquid-acid one.

### Results and discussion

The values of the main properties and characteristics of studied dental cements, resulted from mixtures of solids components (table 2), fired at 1300°C, with a plateau of 4 h and the liquid constituent of the cements, cold prepared at 20°C (table3), are indicated in table 4.

**Standard consistency of cement paste.** In dental technique, depending on the range of the cement and working method used, two consistencies of cement paste

are generally used: for obturations and for fixing prosthetic works [1, 3, 7].

Powder that may be incorporated in a given quantity of liquid determines, in a great measure, the properties of prepared cement mass. Standard consistency of cement paste represents the quantity of cement powder introduced in 0.5 mL liquid, within the period of 3 min and which, under a weight of 120 g, form a disc with the diameter of 30±1 mm. All physical and mechanical characteristics of cements are determined on the paste of standard consistency (according SR 1518/87). Usually, the quantity of powder mixed with 0.5 mL liquid, in order to give a paste of standard consistency, varies for each cement, depending on use, between 1.0 and 1.5 g [8].

The consistency of cement pastes is a measure of their workability, in consonance with the reactivity of basic solid with liquid acid - components of zincophosphate cement. The increasing of solid component basicity, adequately sintered, determines an increasing of the reactivity of the two components of the binder (in the limits of some average values) and reverse - at its decreasing, followed by corresponding increasing or decreasing of standard consistency, respectively. As result from figure 1, together with the data from the tables 2 and 4, increasing of MgO content in the presence of Al<sub>2</sub>O<sub>3</sub> and CaO leads to an increase of cement paste plasticity. SiO<sub>2</sub> presence, in proportion of 1% and over contributes to standard consistency decrease.

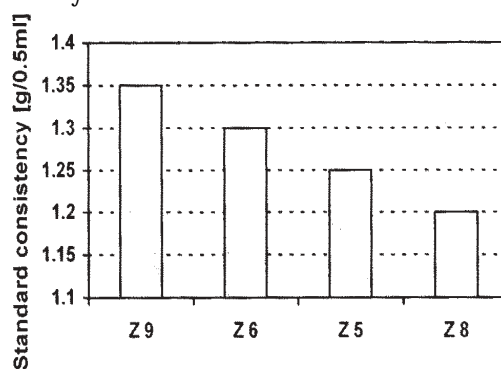


Fig. 1. Influence of solid component composition on standard consistency

Standard consistency decreases below 1g/0.5 mL, for zincophosphate cements which powdery solid component was thermally treated at a temperature under 1300°C (insufficient adequate sintering), for which cooling was made suddenly (fig. 2).

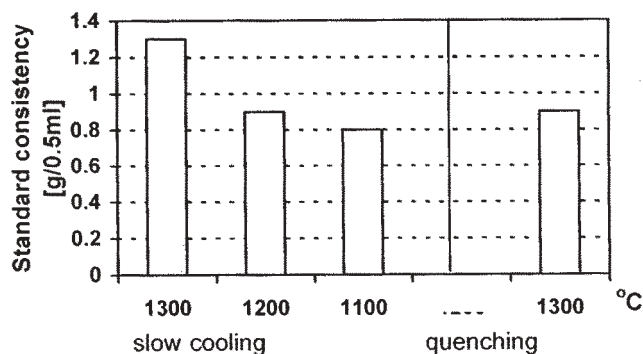


Fig. 2. Influence of solid component sintering parameters on standard consistency for cement Z<sub>6</sub>.

Concerning the dependence of standard consistency on preparation conditions of the liquid component of cement (at 80-100°C or at 20°C), it is stated from figure 3 that this value is higher when a liquid prepared at normal temperature (20°C) is used. This influence is dependent on cement composition (solid component, respectively).

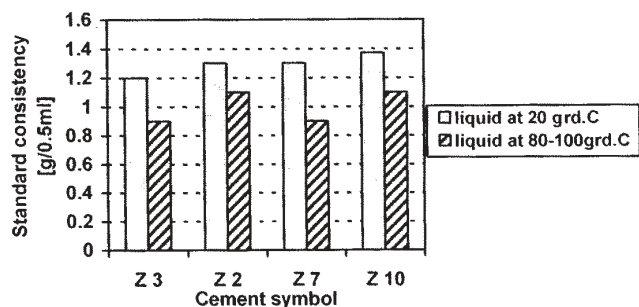


Fig. 3. Influence of the preparation temperature of liquid component of zincophosphate cement in dependence on its solid component composition

The consistency of zincophosphate cement paste is also conditioned by the temperature at which the paste is used. Taking as measure of paste consistency its viscosity, it is remarked that this increases as fast as the time from preparing is higher. Therefore, the using of the paste immediately after preparing is imposed. A paste too viscous cannot penetrate in dental cavity irregularities and consequently the anchoring and the adherence of hardened cement on dental tissues will be damaged; such a contact area will be more vulnerable at organic acids and bacteria medium from mouth cavity.

**Film thickness of binder paste.** This feature has the significance of cement film which is interposed between dentine and the prosthetic work that must be of a few microns (no more than 40 μm). The paste film must be thin enough thin not to modify sensible the space dimensions when the work must be fixed and at the same time to assure a continuous and hard connection between the tooth tissue and prosthetic work.

The method through which the film thickness was determined was according to the Romanian standard STR 1518/87. Influence fundamental factors of film thickness are cement composition and fineness of its powder component (the reactivity of the two components, respectively), paste consistency (viscosity), temperature at which the cementing is achieved, the value and the time of pressure in cementing period [1, 3, 7].

For cement Z<sub>6</sub>, it was found that for Blaine specific surface increase of solid component, from 2000 cm<sup>2</sup>/g to 2500cm<sup>2</sup>/g, the thickness of paste films decreases from 25 μm to 20 μm; at the same time, the cement setting time decreases from 7 to 4 min. Pastes consistency, considered through the ratio powder/liquid, determines at its increase increments of paste film thickness, especially for values over those corresponding to standard consistency - Figure 4. Increase of temperature also leads to paste consistency increase and corresponding consequence on the film thickness of dental cement.

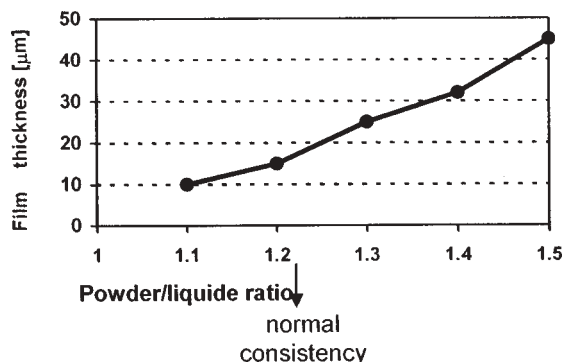


Fig. 4. Thickness film variation with powder/liquide ratio, corresponding to zincophosphate cement Z<sub>6</sub>.

**Setting time.** This is a characteristic of dental cements, resulting from operating requirements and the consequences on hardening process and on mechanical, physico-chemical and structural properties of hardened cement, respectively. Setting time represents the period of time from the beginning of mixing the two components of dental cement in order to obtain a paste of normal consistency, at a temperature of 37°C, until Vicat needle does not let any visible trace on the sample (the first 90 seconds from the setting time represents mixing time).

If the setting is too fast, significant crystallization of hydrophosphate - Zn<sub>3</sub>(PO)<sub>4</sub>·4H<sub>2</sub>O, begins even from the mixing period of the cement components - powder solid and liquid, following within the period of paste introduction in dental cavity. On these conditions, forming the concretion contacts is perturbed and development of crystallization and crystallization-polycondensation structures are also perturbed; thus a mass of low cohesion, friable, results [2, 4, 10, 11]. When the setting time is too long, the intervention is useless prolonged and resistant structure forming is delayed [2, 10, 11].

The results of our work presented in the paper put into evidence the following significant influence factors on setting time:

(i) intrinsic-compositional factors (oxide and phase composition of cement solid component, chemical composition and degree of neutralization of liquid component, respectively) and also the dispersion factors (specific surface area of solid-powder component) and liquid component dilution (water proportion);

(ii) extrinsic factors -operation factors (sintering temperature, powder/liquid ratio, mixing temperature, mixing way of the two components of dental cement and also the way of storage of liquid component).

By examining the information given in the tables 2 and 4, it is remarked that the dependence of cement oxide composition evolves in same way for setting time of zincophosphate cements and for standard consistency, respectively. This finding has the same motivation for obtained setting time values as those given in the case of standard cement pastes consistency. An illustration of this assessment is presented in figure 5.

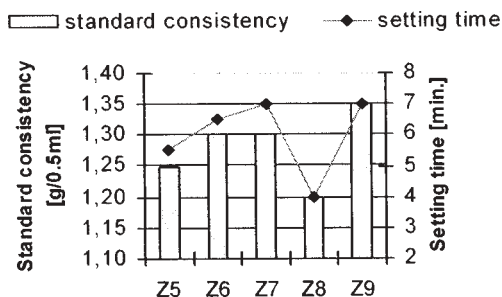


Fig. 5 Influence of solid component composition of studied cements on setting time and standard consistency

Sintering temperature decreasing, when maintaining for 4 h at maximum temperature, leads to a significant decreasing of setting time, when the modification of this temperature is achieved values higher than 1200°C (fig. 6). At the same time, the plateau period at maximum sintering temperature, for an adequate value of setting time is of about 4 h (fig. 7). Rapid cooling in air, after sintering of solid component of the cement  $Z_6$ , leads to setting time decreasing, from 6 ½ min to 3 ½ min.

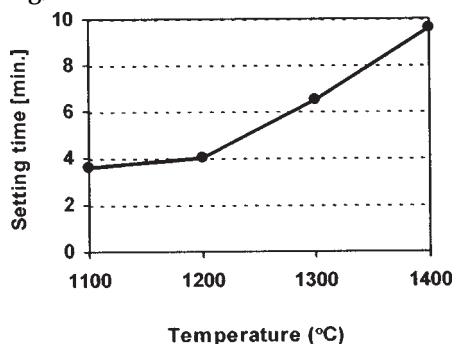


Fig. 6. Influence of powder sintering temperature, component of cement  $Z_6$ , on setting time of this cement.

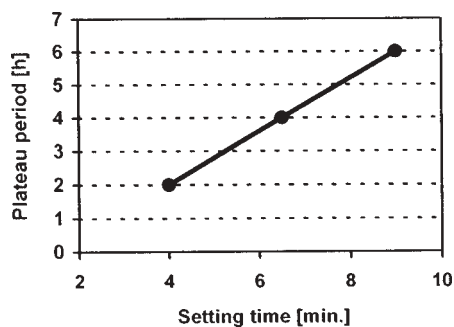


Fig. 7. Influence of sintering plateau period at the temperature of 1300°C for solid component of the cement  $Z_6$ .

In figure 8, the influence of grinding fineness for solid component of dental cement on setting time is presented; as measure of grinding fineness it was considered both dimension of the mesh sieve through which the solid ground component of zincophosphate cement was completely passed, (fig. 8a) and its specific surface area (Blaine) (fig. 8b). It is stated that, for a convenient behaviour of the cement obtained from setting time point of view, the ground solid component of cement must have a Blaine specific surface area of 2000-2500  $\text{cm}^2/\text{g}$ , to pass completely through a sieve of 16000  $\text{mesh}/\text{cm}^2$ - as it has been observed to the studied cements, cement  $Z_6$ , particularly.

Concerning the preparation way of the liquid component (at 80-100 or 20°C) and implicitly its composition, from the histogram of figure 9, it is concluded that the setting time is shorter when using cold prepared liquid, depending on cement nature; for the cements

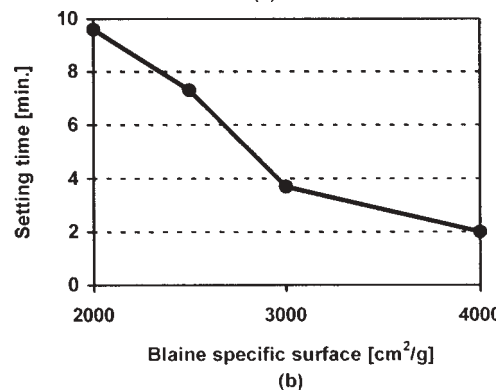
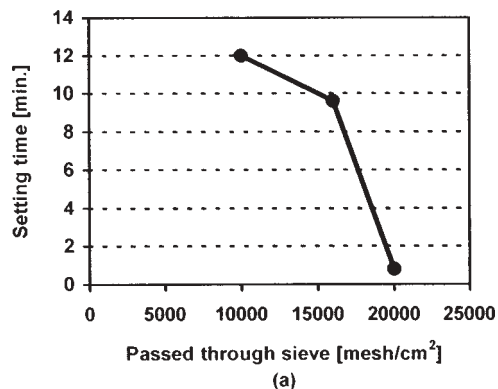


Fig. 8. Influence of grinding fineness of zincophosphate cement ( $Z_6$ ) ground solid component on setting time

considered in figure 9, the setting time values are ranging between 4½ min and 7½ min, being convenient of an adequate casting.

Water content from liquid component influences significantly the setting time. Considering a given content of water-as reference, decreasing its proportion in liquid component (exp.  $Z_6$ ) with two percents leads to prolonging of setting time with 4 min, while water content increasing also with 2 percents leads to setting time shortening, slightly beyond 4 min (fig. 10).

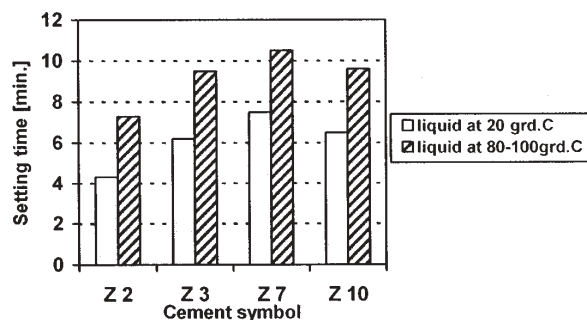


Fig. 9. Influence of liquid preparing conditions on setting time of zincophosphate cements

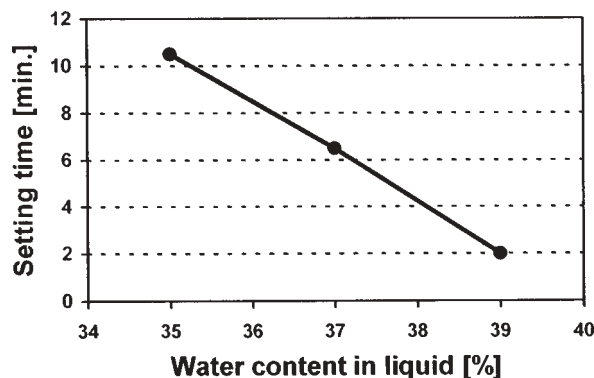


Fig. 10. Effect of water content from liquid component on setting time of cement  $Z_6$

**Table 5**  
MEASURED pH FOR HARDENED ZINCOPHOSPHATE CEMENTS  
AT DIFFERENT TIME PERIODS OF HARDENING

Symbol (cement denomination)	pH for hardened zincophosphate cements at different time periods of hardening				
	3 min.	1 h	24 h	48 h	7 days
Adhesor	4.95	5.30	5.95	6.05	6.15
Phosfodent	4.70	5.10	5.70	5.95	6.01
Z <sub>2</sub>	4.80	5.20	5.90	6.00	6.12
Z <sub>3</sub>	4.80	5.25	5.85	6.01	6.12
Z <sub>6</sub>	5.20	5.45	5.96	6.15	6.20

**Table 6**  
pH OF ZINCOPHOSPHATE CEMENTS COMPONENTS

Analyzed sample	pH
Adhesor powder	9.40
Adhesor liquid	1.55
Phosfodent powder	8.50
Phosfodent liquid	1.80
Z <sub>6</sub> cement powder	9.50
Z <sub>6</sub> cement liquid	1.97

The setting time modification by changing the powder/liquid ratio (keeping unchanged all others significant influence factors) determines- when this ratio is decreased, setting time increase and vice versa. The increase of the setting time by decreasing the powder/liquid ratio must be avoided, as it will unfavorably change other specific properties of the cement.

#### Acidity - pH of the zincophosphate cement paste

The pH values of the dental materials, in general and in particular of the cement pastes have a special importance because these values may influence in a negative way the biological processes of the dental pulp. It is desirable that these materials should have a neutral pH. To the application of the cement paste that has an acid pH, the tooth tissue strives to participate to the acidity temporization, releasing calcium ions (the hydro apatite is stable at a pH ≥ 5.5). Such a process weakens the dental tissues and makes them vulnerable to a future attack. If the dentine layer is thin, there exists the penetration danger of the non-reacted acid from the cement paste, leading to the tooth irritation and degradation and finally will compromise the tooth. This may be avoided by protecting the dental cavity with thin layers (films) of a protection material, such as suspensions of calcium oxide or zinc oxide, zincophosphate cement, zinc oxide - eugenol acid or lacquers based on resins [1, 3]. During cement hardening the pH increases such as, after 24 h, the pH value is ≥ 5.5 stabilizing the dental tissues vis-à-vis the hardened paste. In table 5, there are presented the pH values, for different time periods considered from the 2 components mixing of some studied zincophosphate cements (Z<sub>2</sub>, Z<sub>3</sub>, Z<sub>6</sub>) and reference commercial cements form Czech Republic (Adhesor) and Romania (Phosfodent), as well. The pH tests for the hardened cements were made with a HV-85 pH-meter, on a 2.5 g of hardened cement suspension, in 100 mL distilled water. The Z<sub>6</sub> cement presents a higher pH than the reference cements (table 5).

In table 6, there are given the pH values of the zincophosphate cements components considered in the pH measurements (table 5). The information from table 6 explains why the Z<sub>6</sub> cement develops, for the same time periods, higher pH values than the Adhesor cement that is superior from this point of view to the Phosfodent cement. The explanation consists in a higher reactivity of its solid component with the corresponding liquid component.

The way of liquid preparation, at 80° - 100°C or at 20°C, with consequences over the water proportion and phosphoric acid concentration, is shown for some of the studied cements in figure 11. It is noticed a natural decrease of pH, related with the increasing of the liquid preparation temperature.

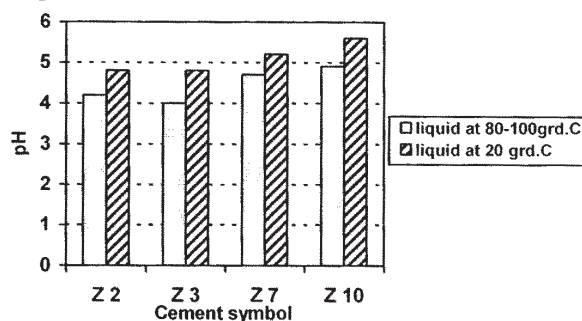


Fig. 11. Influence of the liquid preparation temperature over pH of zincophosphate cements

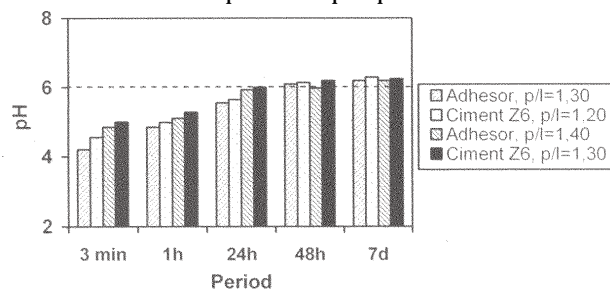


Fig. 12. Influence of cement paste consistency on pH variation in time

At cement paste preparation in the dentist cabinet, it is observed that the cement paste pH is influenced by temperature; the temperature increase accelerates the kinetics of reactions between the solid and liquid components (acid - base reactions), with pH increase. Consequently, at the temperature of the mouth cavity (~37°C) the pH is with 2 units higher than at 20°C.

The increasing of the powder/liquid ratio from the prepared cement paste, by intensifying of the liquid component consumption, obviously leads to the cement pH increase (fig. 12). It is to be seen from figure 12, that for the two considered cements (the cement Z<sub>6</sub>, and Adhesor cement - as reference), a closing of the pH values with hardening time increasing, becoming significant after 48 h (practically equal).

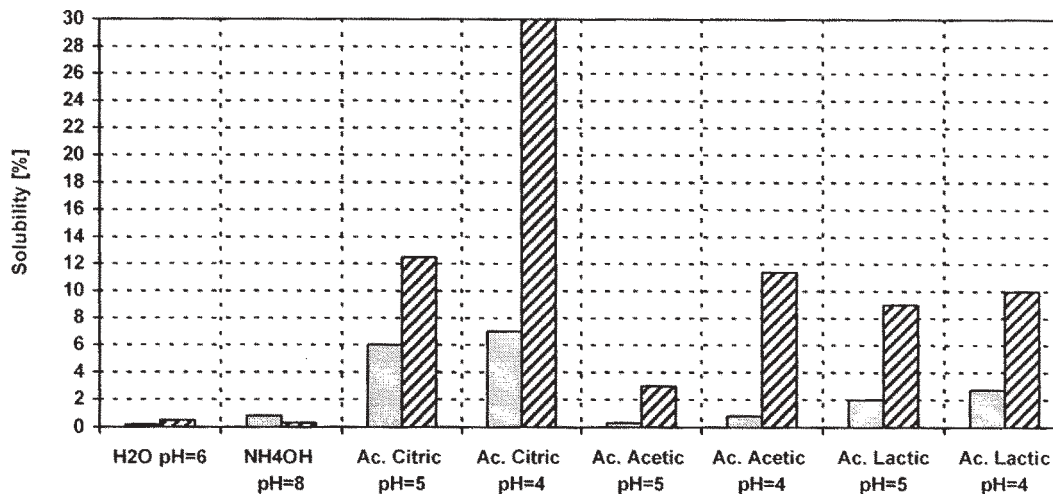


Fig. 13. Zincophosphate cement solubility  $Z_6$ , in different aqueous solutions, after 7 days of immersion

**Solubility and disintegration.** The anhydrous solid component of zincophosphate cements presents a higher solubility than the solid-liquid reactions products (base-acid) – components of these elements. Consequently, in setting period, zincophosphate cement must avoid the contact with water or aqueous solutions; in this stage, solubility in water (aqueous solutions) is high and cohesion of cement paste is lower. After hardening, when hardened resistant structures of the oxide-acid reaction products are developed, constituents are more stable in contact with water, the solubility of binder paste being of lower importance. Cement solubility is strongly influenced by of pH liquid in which this is immersed – the same or periodically changed. In figure 13, the cement solubility  $Z_6$  immersed in different aqueous solutions, for 7 days – with daily changing of these and without changing is presented. The solubility depends on: solute nature from used solutions, pH of aqueous solutions of different substances (particularly acids) and also storage way (with or without daily changing of immersion solution); practically, in all mentioned cases, notable modifications in cement solubility value are recorded.

ADA specification no.8 [8] specifies an admissible maximum cement weight loss, of 0.2 %, by solubilization, at immersion of two discs with  $\varnothing = 20$  mm and local surface of about 1308 mm<sup>2</sup> for 24 h, in distillate water, at 37°C. This method was also applied for testing studied cements in present paper, this being also adopted in Romanian standard SR 1815/87.

Following the powder solid composition influence (see table 2) – component of the cements studied by selection, on cements solubility (fig. 14), it is concluded that in comparison with the cement  $Z_1$  (ZnO-MgO), an addition of 2.50-3.0% MgO, 1% SiO<sub>2</sub> and 2% Al<sub>2</sub>O<sub>3</sub>, leads to sensible decreasing of cements solubility  $Z_6$  and  $Z_7$ ; also adding CaO (1%), together with MgO and Al<sub>2</sub>O<sub>3</sub> but less SiO<sub>2</sub> – in case of cements  $Z_9$  and  $Z_{10}$ , a more decreased solubility is obtained, negligible practically for cement  $Z_{10}$ , that also contain 0.06 % CaF<sub>2</sub> (especially used as fluxing agent).

Solubility of zincophosphate cements is also decreasing with increasing of powder/liquid ratio, up to a certain limit (fig. 15).

Cold preparation of liquid component is favorable to a lower solubility of zincophosphate cement, fact illustrated by the histogram presented in figure 16.

Comparative with zincophosphate reference cement – Adhesor, zincophosphate cements studied in present paper, with MgO, SiO<sub>2</sub> (<1%), Al<sub>2</sub>O<sub>3</sub> (2%), also including CaO (1%) or with CaO (1%) and CaF<sub>2</sub> (0.06 %), are

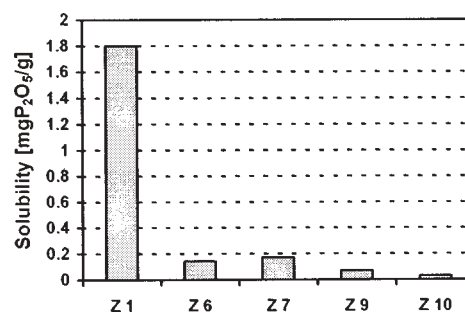


Fig. 14. Solid component composition influence of studied cements on their solubility

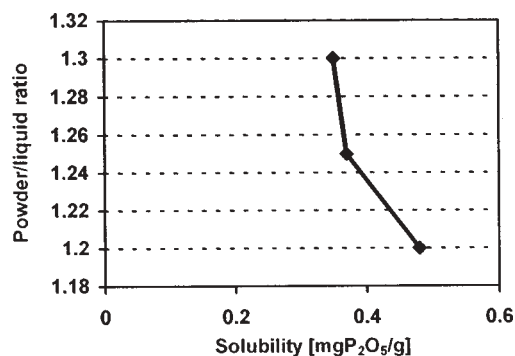


Fig. 15. Powder/liquid ratio influence on zincophosphate cement solubility  $Z_3$ .

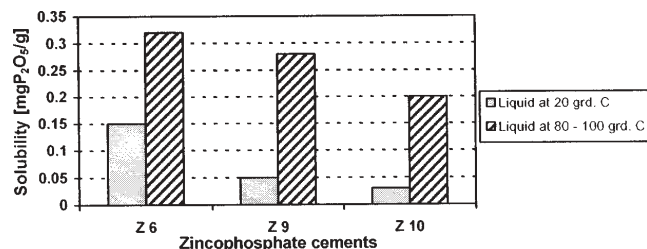


Fig. 16. Influence of preparation temperature of liquid component of zincophosphate cement on its solubility

characterized by low solubilities, especially in the case of cements  $Z_9$  and  $Z_{10}$  (fig. 17).

**Mechanical strengths.** Mechanical properties of zincophosphate cements are assessed through their compression strength. Compression strength of these cements is essentially conditioned by their solid component – at a given composition of used liquid component, powder solid/liquid ratio, mixing mode of the

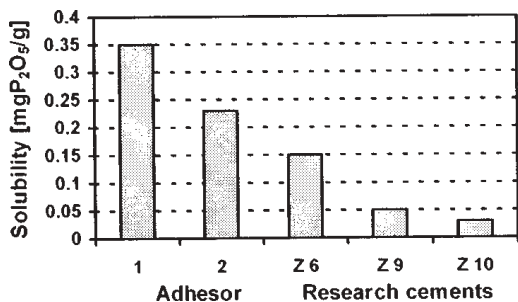


Fig. 17. Comparative histogram of solubilities for Z<sub>6</sub>, Z<sub>9</sub> and Z<sub>10</sub> cements, with solubilities of reference cements Adhesor (1 and 2)

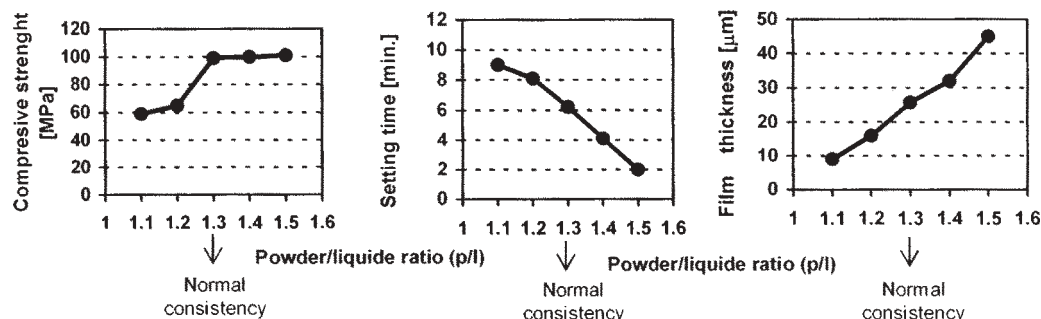


Fig. 18. Powdery solid/liquid ratio influence on compression strength, setting time and thickness of film cement paste Z<sub>6</sub>.

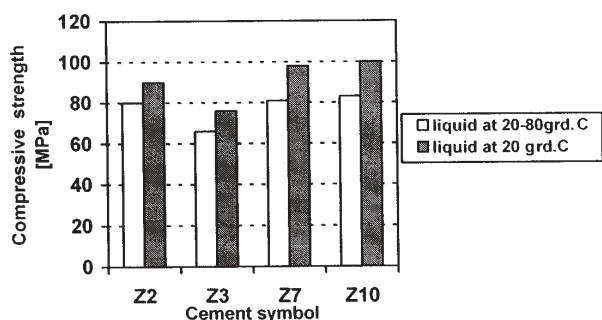


Fig. 19. The influence of preparation condition for liquid component of zincophosphate cement on compression strength

components (for a high homogenization of paste and without water loss from liquid component or from paste) and casting (with isolation, after case, of work area from mouth cavity, during setting time, up to paste hardening).

Minimum strength after 24 h of hardening is, in accordance with different standards, of 70-75 MPa. Hardening structure and compression strength for zincophosphate cements are rapidly developed at least 2/3 from the final strength, after only one hour from mixing the two components - solid and liquid.

Analyzing the information given in tables 2 and 4, it is stated that from chemical composition of powder solid point of view, constituent of zincophosphate cements - sintered and ground accordingly, it might be concluded that, when increasing the MgO content up to 8.50 % - in presence of SiO<sub>2</sub> (1%), Al<sub>2</sub>O<sub>3</sub> (2%) and Ca (1%), respectively, together with a relative small proportion of CaF<sub>2</sub> (0.06%), the resistance increases, in some cases, remarkably (see series of cements Z<sub>2</sub>, Z<sub>6</sub>, Z<sub>7</sub>, Z<sub>9</sub> and Z<sub>10</sub>).

The ratio powder solid/liquid presents an obvious positive influence on compression strength of zincophosphate cement, when the standard consistency value (normal) is reached; over this value, cement strength, at 24 h of hardening, is not changed. This estimation is illustrated, for the cement Z<sub>6</sub>, in figure 18, comparative with the correlations between the setting time, thickness of film from binder paste and the ratio powder solid/liquid of the same cement. If for the values of paste consistency higher

than standard consistency value, it was found that mechanical strength presents negligible increasing, for the setting time a decrease is noted and for paste film thickness - relevant increasing. This behaviour represents consonant effects of reactivity between the two components of cement: solid (base) - liquid (acid).

Influence of liquid preparing temperature (at 80°C - 100°C or at 20°C), in correlation with zincophosphate cements composition, is illustrated in figure 19. It is remarked for all considered cements the lower mechanical resistances in the case of liquid preparation at higher temperatures. This statement is a consequence of water loss, followed by acidity increasing, with inadequate intensification of acid-base reaction kinetics; in these conditions, hardening structures to be formed are tensioned having as effect the compression strength decrease.

## Conclusions

The obtained results and their interpretation lead to conclusions of fundamental nature regarding the preparative chemistry basis of zincophosphate cements, in correlation with casting and functional requirements.

-Zincophosphate cements are binders with fast hardening. Consequently, adequate moderation of their solid-basic component reactivity with that of liquid-acid has a deciding importance for the obtained cement quality; it must be solved in consonance with the requirements when using these cements - operational and functional. The mentioned reactivity is dependent on the composition and processing the parameters of the two cement components.

The reactivity of the two components of zincophosphate cements, together with processing parameters at casting, is determinant for characteristic properties of corresponding dental cements.

- The parameters for processing of the two components of zincophosphate cements are sintering conditions and grinding fineness - for powder solid, neutralization degree, water ratio and preparing temperature, respectively - for liquid component. Thus, these are also determined by taking into account the chemical composition of each components, phase component, basicity and dispersion



parameters –for powder solid component reactivity, phase composition respectively, and acidity and liquid component concentration –for the latter component of zincophosphate cement.

- Processing parameters at casting of zincophosphate cements – powder/liquid ratio, preparing way of liquid phase and cement paste (components mixing, temperature of the pastes, storage way of liquid component), are added to the other influence factors – compositional and processing, in obtaining the two components of the cements, in order to achieve the reactivity of the last ones. In this way – direct or indirect, there are also adjusted, after the case, the characteristic properties of the cements – in operation and functioning.

- In our study correlations composition-processing-properties are emphasized, establishing the influence factors determinant for zincophosphate cements properties. Mechanisms of reaction processes and hardening of these cements were suggested. Dental cements with relevant properties were obtained.

### **Bibliography**

1. SKINER, E.W., PHILLIPS, R.W., "The science of dental materials". Ed. VI-a Philadelphia and London, 1976

2. GOLĂNKO-VOLFSON, S.L., SÎCEV M.M., SUDAKAS, L.G., SKOBLO, L.I., "Himiceskie osnovitehnologii i primeneniia fosfatrîh sviazok i pokrîtii". Izd. Himia, Moskva, 1968
3. CRAIG, R., Restorative dental materials, St. Louis, Baltimore, Toronto, 1989
4. TEOREANU, I., DUMITRESCU, C., "Zincophosphate ciments – hardening mechanisms". Rev. Roum. Chim., **47**, nr.3-4, 2002, p 393
5. GEORGESCU, M., PURI, A., Chemistry of inorganic binders (in Romanian), Editura Politehnica-Press, Bucuresti, 2004, p. 400
6. WILSON, A.D., NICHOLSON, J.W., Acid base cements - their biomedical and industrial applications. Cambridge, 1993
7. DUMITRESCU, St., COCA, I., "Materials used in dentistry (in Romanian)", Editura Pedagogică, Bucuresti, 1979
8. ADA nr. 8, Dental Zinkphosphat cement, 1980, USA
9. NORMAN, R.D., SWARTZ, M.L., PHILLIPS, R.W., "Studies on film thickness, solubility and marginal beakage of dental cements, Dent. Res.", **42**, 1963, p. 950
10. WILSON, A.D., "The chemistry of dental cements, Chem. Soc. Rev.", **7**, 1978, p. 265
11. DUMITRESCU, C., "Special binders with hardening based on acid-base reactions (in Romanian), Ph D Thesis. University POLITEHNICA, Bucharest, Romania, 1998

---

Manuscript received: 9.03.2007











