

Journal of Silicate Based and Composite Materials

A TARTALOMBÓL:

- Strengthening of masonry walls with CFRP composite: experiments and numerical modeling
- Evaluation of index and compaction properties of lateritic soils treated with quarry dust based geopolymer cement for subgrade purpose
- Studying compression strength of XD3 concrete samples after addition of calcium nitrate inhibitor and superplasticizers
- Material removal characteristics of AI-SiO₂ composite in WEDM
- Superficial hardening improvement of nano and micro composite AI TiC
- Creation of "necessary" mixtures of baking soda, hydrogen peroxide and warm water as a strategy for modernization bleaching cleaners of ceramic

2020/1





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2020/1

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A finomkerámia-, üveg-, cement-, mész-, beton-, tégla- és cserép-, kő- és kavics-, tűzállóanyag-, szigetelőanyag-iparágak szakmai lapja Scientific journal of ceramics, glass, cement, concrete, clay products, stone and gravel, insulating and fireproof materials and composites

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Strengthening of masonry walls with CFRP composite: experiments and numerical modeling

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Abstract

The paper presents the experimental and numerical study of brick masonry walls reinforced with carbon fiber-reinforced polymer (CFRP) composite subjected to a shear load. In this analysis the Concrete Damaged Plasticity (CDP) constitutive laws are utilzed to simulate the non-linearities in the behavior of brick and mortar. These models are used for the analysis of diagonal compression tests on masonry panels. The experimental results are used to validate the modelling approach presented here. The results showed that the application of the CFRP composite on unreinforced masonry (URM) has a great influence on strength, post-peak behavior, as well as changing failure modes and ductility. On the other hand, the adopted detailed micro-modelling approach (DMM) gives a good interface for predict the shear response on masonry walls.

Keywords: masonry walls, CFRP composites, shear loads, detailed micro-modelling approach, diagonal compression tests

Kulcsszavak: téglafalazatok, CFRP kompozitok, nyírás, részletes mikro-modellezési eljárás, átlós nyomószilárdság teszt

1. Introduction

Around the world, there are many old unreinforced masonry (URM) structures such as historical cultural monuments and bridges, etc. which are deteriorated or damaged during earthquake events [1, 2]. In order to extend the life of such structures, strengthening or repairing by implementing new techniques have been developed. Many of these strengthening techniques including the use of fiber reinforced polymer (FRP) composites were used to reinforce masonry structures. These composites are manufactured in different features depending on the fiber material type like carbon fiber (CFRP), glass (GFRP), and aramid (AFRP). FRP strips offer the possibility of application by gluing on the outside surface EB (externally bonded) or inserting inside the groove of element by the near-surface mounted (NSM)technique. The use of FRP composites embedded into polymetric matrix such as (polyester, epoxy, mortar mix) can be the perfect solution for retrofit the structure walls in seismic area. The application of FRP composite to strengthen and rehabilitate masonry has been studied by several authors. Experimental studies evaluate the effect of different variables for example, the effect of retrofitting configuration of FRP composite, type of FRP composites and the type of masonry components, through small-scale testing such as triplets test, Wallette and by full scale testing. Mahmood and Ingham [3] conducted a series of experimental test on URM walls, they illustrated the effectiveness of FRP retrofitting system in improving the shear strength of unreinforced masonry with a factor of 3.25. Valluzzi et al [4] tested a series of unreinforced brick masonry panels strengthened by different materials (GFRP, CFRP and PVAFRP) of different configuration. All tested specimens were subjected to a diagonal compression test, the results showed that the application of FRP at one side of the wall only produces a significant out-of-plane deformation, corners and the failure mode of the out-of-plane displacement exaggerated by the little restraint at the top and bottom of the wall. Several researchers such as Haroun et al [5], Tumialam et al [6], Hamid et al [7] and Li et al [8] have shown that FRP composites can improve the shear capacities of URM walls significantly. All these experimental studies have shown that the use of FRP strengthening technique can ensure adequate increase of seismic capacity, stiffness and ductility of masonry wall in the in plane lateral loading [9,10] or out-of-plane [11].

To understand the structural behavior of the masonry wall during earthquakes, it is significant to model this using advanced technique. In the literature two major approaches have been developed for masonry modeling namely heterogeneous and homogeneous modeling. In the first approach the brick units and joint mortar are considered separately (heterogeneous modeling). While, in the second approach (homogeneous modeling) the brick units, joint mortar and interfaces between them are assumed to be either an isotropic or anisotropic composite material. According to the classification of Lourenco (1995) [12] there are two main modelling approaches: macro-modelling and micro-modelling (see *Fig.1*). The micro-modeling can be divided into two techniques as detailed micro-modeling and simplified micro modeling [13].



Fig. 1Modelling strategies for masonry structures1. ábraFalazott szerkezetek modellezési stratégiái

In the detailed micro-modeling (see *Fig. 1b*) each component (units and mortar) is modeled separately with unit-mortar interface. The concept of micro modeling is adopted for analysis in detail of small masonry structural elements such as masonry shear triplets or small panel wall. In this approach the masonry units and mortar are modelled with real thickness, while zero thickness is assigned to unit-mortar interfaces. The units and mortar are modelled as continuum elements and unit-mortar interfaces are modelled as discontinuous elements.

The literature has been clearly focused on the importance of including all mechanisms of rupture of the masonry in the modeling to understand its behavior, in terms of ductility and ultimate load as well as the damage which is usually concentrated at the mortar interface [14,15]. In microscopic models, only a tensile breaking of the bricks is sometimes considered, most studies relying only on a linear elastic behavior of the bricks. Or in other words, the authors adopted this model assent to the need to introduce the post-peak softening behaviors of the mortar. The progressive cracking of the elements must be reproduced by the softening in tension. In shear, the degradation of the cohesion and that in compression leads to the crushing of the masonry. The softening behavior laws, which are thus essential in modeling, however, rely on the definition of many parameters whose values are difficult to be predicted. This modeling is therefore only feasible if it is coupled with a several experimental test for best description of the material and their interactions.

In the present study, the influence of the type of joint mortar and location of the CFRP composites in the strengthened panels subjected to shear loads is evaluated by diagonal compression test. For the numerical analysis, a numerical model based on the DMM approach was developed using FE software ABAQUS and validate with the experimental data. The result of these numerical examples is compared with the experimental result.

2. Experimental program

The test consists of testing four unreinforced walls (control panels noted MT) and six walls reinforced with CFRP composites in order to evaluate the reinforcement efficiency and the mode of rupture in case of each reinforcement. The reinforced panels were strengthened by Sika Warp carbon fiber CFRP composites of 50 mm width. The FRP reinforcement was glued, using two-part epoxy adhesive. Three configurations of the retrofit system were investigated.

2.1 Materials

2.1.1 Brick units

The perforated bricks were tested in compression and in bending. Five test series has been studied to obtain the average values of compressive strength and the elastic modulus. The tests were carried out using a hydraulic press according to the EN 771-1 [16].

2.1.2 Cement-Lime mortar

The cement-lime mortars are commonly adopted, because these mortars have the good properties of cement as well as lime mortars, that is, medium strength along with good water retentively, good workability and to some extent freedom from cracks. Therefore, this type of mortar has been adopted.

Two mixes consisting of cement: lime: sand proportions (1:1:3 and 1:1:5) were prepared using an electrical mixer by weight batching. Water was added and the mixture was remixed to achieve a workable consistency. In order to measure the mechanical properties of these mortars, the prepared mixtures were cast into standard molds and then maintained in the standard curing. The mechanical properties of mortars were determined at the age of 3, 7, 14 and 28 days after de-molding. Flexural and compressive strength tests were measured according to EN 1015-11 [17]. By using the universal testing machine, the flexural strength has been tested on specimens in the shape of a prism 40×40×160mm³ . Afterward, the two half-prisms obtained after breaking into two parts from the specimen during the flexural test were subjected to the uniaxial compressive test (results are shown in Table 1). Six specimens of each mix are tested to have the average value of the response. The average values of the brick compressive strength, the mortars compressive strength and secant modulus of elasticity are reported in Table 1.

Material	Compressive strength (MPa)	Flexural strength (MPa)	Young's modulus (MPa)
Mortar A (1:1:3)	7.187	3.341	3639.24
Mortar B (1:1:5)	3.643	1.453	1880.87
Brick	20.53	-	1.E+04

 Table 1
 Compressive strength, flexural strength and young modulus of mortar and brick

 1. táblázat
 A habarcs és tégla nyomószilárdsága, hajlítószilárdsága és rugalmassági modulusa

2.1.3 Composite materials (SikaWrap carbon fiber)

In this study, a unidirectional carbon fibre (Sika Wrap carbon fiber fabric) is employed. The technical characteristics are summarized in the following *Table 2*.

Property	Value
CFRP width	300 / 600 mm
CFRP length / roll	≥ 50 m
Weight	$235 \text{ g/m}^2 \pm 10 \text{ g/m}^2$
Thickness	0.129 mm
Density (Fiber)	1.82 g/cm ³
E _{CERP}	230 kN/mm ²
Ft _{CFRP}	4000 N/mm ²
Rupture strain	1.70%

Table 2 Mechanical properties of the CFRP reinforcing system (nominal values reported by the manufacturer)

2. táblázat A CFRP mechanikai tulajdonságai (gyártó által közölt névleges értékek)

2.2 Methods

The test specimens were made according to the instructions given in RILEM technical recommendation [18]. A series of ten masonry wallettes were constructed from perforated brick with dimensions $220 \times 105 \times 55 \text{ mm}^3$ and mortar joint with 10 mm of thickness. Two types of mortar (type A, type B) were used in the construction of the panels having mix ratio of 1:1:3 and 1:1:5 of (Portland cement: hydrated lime: sand). The test involves subjecting a square section of masonry, with global dimensions $400 \times 400 \times 105 \text{ mm}^3$, to a compressive load applied along the diagonal. The experimental setup for the diagonal compression test is presented in *Fig. 2*.





Fig. 2 Experimental setup of unreinforced masonry panels tested in diagonal compression 2. ábra Kísérleti összeállítás megerősítetlen falazat diagonális nyomószilárdság vizsgálatára

The walls are placed and centered diagonally between the two plates of the press with the help of the metal shoes. These allow the transmission of the load to the wall in the vertical direction. The load is applied using the hydraulic cylinder which is placed below the load cell. The measurement of displacements is carried out using two displacement sensors which are installed on the diagonal of the wall. All data, forces and displacements, are automatically recorded by a data acquisition system, and given automatically by the system. The loading is applied by a machine of capacity 500 kN. The loading speed is 5 mm/min. The tests were performed under displacement control in order to obtain the complete stress-strain curve of the panels. All tested wall panels were of similar dimensions in order to permit direct comparison of their failure loads (see *Figs. 3, 4*).



Fig. 3 Specimen preparations of unreinforced masonry panels 3. ábra Megerősítés nélküli falazat minták előkészítése

The shear strength is calculated according to the state of stress in the center of the wall; isotropic or anisotropic. The calculation of the shear stress τdt is made according to ASTM 519 - 02[19]. considering a state of isotropic stress in the center of the wall.

$$\tau_{dt} = \frac{P_{\text{max}}}{\sqrt{2 \times A}} \tag{1}$$

The lateral surface A is subjected to a maximum load P_{max} Area A is calculated with Equation (2) by considering w width, h height and t wall thickness.

$$A = \frac{w+h}{2} \times t \tag{2}$$

The displacement ductility factor (μ) is defined as the ratio between the ultimate displacement to the yield displacement:

(3)

 δ_{μ} : displacement at ultimate load

 δ_{y} : displacement at the load causing yield condition

The shear modulus G is calculated in the zone of elastic stress with Equation (4)

$$G = \frac{\Delta \tau_e}{\Delta \gamma_e} \tag{4}$$

 $\Delta \tau_e$ and $\Delta \gamma_e$ represent stress and elastic strain, respectively.



Fig. 4 Specimen preparations and configuration of reinforcing for masonry panels 4. ábra Megerősített falazat minták előkészítése

3. Experimental results and discussion

3.1 Behavior of control walls

In wall panels MTA and MTB cracking occurred predominately through the mortar joints in a diagonal, followed by a rapid decrease in load capacity. With load increasing, the wall exhibited a gradual increase in the width of predominately diagonally oriented crack, with further increase in load multiple cracks were observed in the panel before failure as shown in Fig. 5. The MTA control wall has a maximum shear strength of 0.5 MPa corresponding to a maximum force of 33.66 kN. In addition, the MTB wall has a breaking force of 35.06 kN, which produces in this wall a maximum shear stress of 0.56 MPa (see Table 3). The shear stress-strain response of the tested unreinforced wall panels (MTA and MTB) is summarized in Fig. 7. For a comparison between the two responses, The wall panel constructed with mortar A failed at lower load compared to the wall panel type B,but with a slight difference. Both MTA and MTB wall panels exhibited an approximately linear shear stress-strain response until cracking, for the wall MTA followed by rapid degradation of shear strength once cracking propagated, but for wall MTB followed by a slight increase in shear strength and deformation capacity before the rupture.



Fig. 5 Failure modes of control masonry panels: (a) MTA, (b) MTB 5. ábra A referencia falazat panelek tönkremeneteli módjai

3.2 Reinforced masonry panels subjected to the diagonal compression

In unreinforced walls, the tensile stress causes an appearance of cracks leading to a complete destruction. However, in the case of walls strengthened with CFRP composites, the tensile stresses are transferred to these strips and it results in a significant reduction of stress in the masonry wall. Regarding the overall response of the walls, the results obtained revealed a significant increase in shear stress from 65% to 270% compared to unreinforced walls. Likewise, it was found that the improvement in ductility for strengthened wall panels type A ranged from 74% to 80%, whereas for wall panels type B it ranged between 80% to 88% (see Table 3). The CFRP reinforced panel failed suddenly due to a cracking along the compressed diagonal at the ends of the composite strips (see Fig. 6). The MRX and MRH walls have a maximum force of 55.27 kN and 57.58 kN respectively, which is a value of 1.82 MPa and 1.85 MPa for shear strength. and present better shear behavior with increase of 200%. This reinforcement allowed to increase the ductility µ up to 88%. Thus, when the joint of the mortar cracks, there is a redistribution of the force towards the part of the reinforcement which is in the vicinity of the crack. Therefore, the arrangement of the reinforcing composites has a very important effect on the local behavior of the structure, due to the stress distribution and the deformation of the structure. An application of CFRP composite on a 24% wall surface sufficient to increase wall ductility, and give almost the same results as that recorded when the fabric covers an area of 54 and 56%. The first conclusion, which can be obtained from the experimental results, is that the wall panels reinforced by CFRP composite technique presented more ductile behavior compared with the control wall panels for each type of mortar (see Fig. 7). Moreover, the shear strength of reinforced wall panels is dependent on the mortar resistance.

panels	type	fiber	F _{max} (kN)	τ (MPa)	G (MPa)	γe	γu	μ	(µ _u /µ _。)
MT	А	0%	33.66	0.50	77	0.023	0.027	1.16	
	В	0%	35.063	0.56	78	0.029	0.031	0.94	
MRH	А	54.68%	54.958	1.76	92	0.056	0.111	2.02	73.98
	В	54.68%	57.583	1.85	80	0.033	0.058	1.74	85.06
MRX	Α	54.06%	50.815	1.61	117	0.027	0.057	2.1	80.87
	В	54.06%	55.274	1.82	128	0.028	0.049	1.78	88.83
MRI	Α	22.91%	50.455	1.59	113	0.021	0.039	1.90	64.09
	В	22.91%	45.607	1.45	103	0.021	0.036	1.80	80.00

Ye: Elastic deformation – Yu: Maximum deformation at ultimate load – μ : ductility factor – (μ_u/μ_o): improvement between unreinforced and reinforced masonry walls – fibers (%): percentage occupation of the surface walls by carbon fiber band

 Table 3
 Ultimate shear strength and ductility factors for experimental tests of unreinforced and reinforced wall

3. táblázat A megerősített és megerősítés nélküli falazatok nyírószilárdsága és duktilitási tényezői



- Fig. 6 Failure modes of all strengthened masonry panels: (a) MHA and MHB, (b) MIA and MIB, (c) MXA and MXB
- 6. ábra Megerősített falpanelek tönkremeneteli módjai: (a) MHA és MHB, (b) MIA és MIB, (c) MXA és MXB



 Fig. 7 Shear stress-strain relationship for unreinforced and reinforced masonry panels
 7. ábra Nyírófeszültség-alakváltozás összefüggése megerősítés nélküli és megerősített falpanelek esetén

4. FEM modeling for unreinforced masonry wall (MTB)

4.1 Material parameters

In this study, the FE software, ABAQUS was used to evaluate the validity of proposed model (DMM) approach for predicting behavior of brick masonry wall. The units and mortar joints are modelled using eight noded 3D continuum elements with hour glass control and reduced integration (C3D8R), and the unit-mortar interface with zero thickness. The penalty friction formulation was introduced to model tangential behavior with a friction coefficient value of 0.6. The normal behavior of the interface was modeled using "hard contact", the nonlinear behavior of brick and mortar was simulated by using the CDP model. The interface between unit and mortar was modeled through surface-to surface contact. The adopted modeling strategy used in this study is illustrated in *Fig. 8*.



Fig. 8 Adopted detailed micro-modeling (DMM) approach 8. ábra Részletes mikro-modellezési megközelítés

4.2 Presentation of the numerical model

To validate the model proposed in this study, the same wall that which was studied in the experimental part was chosen (MTB). *Fig. 9* shows the geometry and loading condition for the FE model that has been implemented using ABAQUS.



Fig. 9 Numerical model (DMM) and boundary conditions of unreinforced brick masonry wall

9. ábra Numerikus modell és peremfeltételei megerősítés nélküli falazat esetén

4.3 Constitutive behavior of units and mortar

4.3.1 Concrete damage plasticity (CDP) model

The Concrete damage plasticity (CDP) model provides a general capability for modeling concrete and other quasibrittle materials. This model which was developed by Lubliner et al (1989) [20] and adopted by Abaqus is selected in this study to simulate the nonlinear behavior of the masonry units. This approach has been developed to predict the two main masonry failure mechanisms such as cracking under tension and crushing under compression. The CDP model uses the concepts of isotropic damaged elasticity in combination with isotropic tensile and compressive plasticity [21]. The level of damage is represented by damage parameters d_c and d_t as shown in *Figs. 10* and *11*.



Fig. 10 Brick and mortar response to uni-axial loading; (a) Brick: in tension and compression; (b) Mortar: in tension and compression [19]
10. ábra Tégla és habarcs viselkedése egytengelyű terhelés hatására: (a) tégla: húzás és nyomás; (b) Habarcs: húzás és nyomás [19]

4.3.2 Constitutive behavior of units-mortar interface

In this model, brick-mortar interface is modeled by means of Coulomb friction criterion, in order to adequately reproduce the shear response of joint, the values of cohesion and friction angle for a Coulomb type friction model are provided by the envelop of the shear strength for different normal stress values, which was obtained experimentally by the triplet test (see *Fig. 12*). The interaction module of Abaqus/Explicit analysis was used to make the contact between units and mortar through the option surface-to surface contact. In this step it is necessary to define two contact properties: normal contact and tangential behavior.

4.4 Model input parameter

In this section, the input parameters used to reproduce the mechanical behavior of masonry wall using the detailed micromodeling approach is presented. The mechanical parameters for brick and mortar employed in this analysis were obtained from tests carried out in this research (see *Tables 4*, *5*).

Elasticity parameters	Brick	Mortar
Mass Density γ (kg/m³)	2200	1800
Young's modulus (MPa)	10000	1880
Poisson ratio v	0.2	0.18

 Table 4
 Mechanical properties of masonry unit and mortar

4. táblázat A habarcs és a falazat mechanikai tulajdonságai

Plasticity parameters						
Dilation angle $\Psi(°)$	20					
Eccentricity parameter e	0.1					
Biaxial and unidirectional initial compressive strength ratio	1.16					
Stress ratio in tensile meridian K	0.67					
Viscosity Parameter	0.0001					

 Table 5
 Concrete damage plasticity of masonry brick and mortar

 5. táblázat
 A falazat képlékenyedési paraméterei



Fig. 11 Brick and mortar response to uni-axial loading; (a) Brick: in tension and compression; (b) Mortar: in tension and compression (present work)

11. ábra Tégla és habarcs viselkedése egytengelyű terhelés hatására: (a) tégla: húzás és nyomás; (b) Habarcs: húzás és nyomás (kísérletek alapján)



Fig. 13 Confrontation of curves $(\delta - \varepsilon)$ 13. ábra Görbék összehasonlítása $(\sigma - \varepsilon)$

The shear characteristics of the masonry and the brick/ mortar joint interaction parameters at the interface are determined on a masonry prism (triplet), the test results of 16 triplets for confining stress varying from 0 to 1 MPa allowed us to determine the parameters shear strength and friction coefficient.

The shear strength (initial shear strength without any vertical stresses) is determined as follows:

$$\tau = \frac{P_{max}}{2A} \tag{5}$$

Where P is the shear load at failure and A is the cross-sectional area of contact between two bricks.



Fig. 14 Von Mises stress distributions, deplacement in the direction y (U₂₂), plastic strain distributions (PEEQ) in the unreinforced brick masonry wall
14. ábra Von Mises feszültség eloszlás, y irányú elmozdulás (U₂₂), képlékeny alakváltozás (PEEQ) megerősítés nélküli tégla falazat esetén



Fig. 15 Evolution of damage (DAMAGET, DAMAGEC) in the unreinforced brick masonry wall 15. ábra Tönkrementel kialakulása (DAMAGET, DAMAGEC) megerősítés nélküli falazatokban

The shear strength of the unit-mortar interface τ of masonry under normal stress was characterized through Coulomb failure criterion $\tau_u = c + \mu * \sigma_n$ (6)

Where $\mu = tan\varphi$ and $c=\tau_0$ are the coefficient of friction and cohesion respectively.

The damage plasticity constitutive model was based on the following stress-strain relationship:

$$\sigma = (1 - d_t)\overline{\sigma}_t + (1 - d_c)\overline{\sigma}_c \tag{7}$$

where d_c and d_t were two scalar damage variables, ranging from 0 to 1.

the damaged parameter (d_c) is calculated by

$$d_{c} = 1 - \frac{\sigma_{c}}{\sigma_{c}} \tag{8}$$

Where σ'_{c} is the compressive strength of masonry.

Damaged parameter (
$$d_t$$
) can be calculated by equation (9)
 $d_t = 1 - \frac{\sigma_t}{\sigma_{t-1}}$ (9)

where σ'_t is the masonry tensile strength

The uniaxial compressive and tensile responses of concrete with respect to the concrete damage plasticity model subjected to compression and tension load were given by:

$$\sigma_t = (1 - d_t) E_0 (\varepsilon_t - \tilde{\varepsilon}_t^{pl}) \tag{10}$$

$$\sigma_c = (1 - d_c) E_0(\varepsilon_c - \tilde{\varepsilon}_c^{pl}) \tag{11}$$

the effective uniaxial stress σ_t and σ_c were derived as follows:

$$\bar{\sigma}_t = \frac{\sigma_t}{(1-d_t)} E_0(\varepsilon_t - \tilde{\varepsilon}_t^{pl}) \tag{12}$$

$$\bar{\sigma}_c = \frac{\sigma_c}{(1-d_c)} E_0(\varepsilon_c - \tilde{\varepsilon}_c^{pl}) \tag{13}$$

4.4 Comparison of results and discussion

The response obtained from the model is illustrated by the stress-strain diagram and by the damage of the panel. *Fig. 13* shows the numerical and experimental curves of the stress-strain relationship of unreinforced walls (MTB). The numerical results show a good agreement with the experimental results concerning not only at the initial rigidity of the elastic phase but also from the non-linear phase to the post peak response corresponding, but with a value from the numerical stress to the higher peak than the experimental value. Through these results show that the technique proposed in this study gives good results to analyzise the brick masonry wall behavior.



Fig. 16 Comparison between the numerical and experimental results concerning the crack pattern for the unreinforced wall MT: (a) experimental crack pattern; (b) numerical crack pattern
16. ábra Numerikus és kísérleti eredmények összehasonlítása a megerősítés nélküli MT falazat törésképét kiemelve: (a) kísérleti eredmény; (b) numerikus eredmény

4.4.1 Crack pattern and mode failure

Figs. 14 and 15 show the initial cracking occuring along the bed and head mortar joints in a diagonal (initial step). As the vertical loads increase, more cracks occur in the mortar joints of wall from top to down. After that, cracks appear and propagate in the brick directly in final step. Same mode of rupture was found experimentally.

Fig. 16 shows a comparison of the crack pattern developed in the numerical and experimental test for the masonry wall MTB. The crack patterns observed in the mortar joint and brick during the experiment test and predicted by FE model resemble each other to a good extent. A good correlation was found not only at the crack pattern of mortar but also from the brick. However, the position of cracks at the brick in numerical case differs from experimental test, this can be explained by the numerical simplification which consists in considering that all the mortar joints have the same thickness, the same mechanical characteristics, which is not assured experimentally.

5. Conclusions and future work

There is significant potential for the application of FRP in the masonry industry, both in the construction and rehabilitation of older structures. In this paper, the behavior of brick masonry wall reinforced with externally bonded of CFRP composites is studied with experimental and numerical investigations. The obtained experimental results have been validated by a new modeling technique based on the DMM approach. The following conclusions were drawn from the experimental and numerical study:

 the increase in the proportion of sand in the mortar (from 3 to 5) led to an increase in the shear strength and the ductility of the masonry panels, especially in the case of reinforcement.

- The improvement in shear strength for strengthened wall panels with CFRP composite ranged from 3 to 4 times (65% to 270%).
- All reinforced wall panels showed a substantial increase in the ductility by 73% to 88% compared with unreinforced wall panels.
- The most significant increase in ductility was achieved by reinforcement in both sides of the wall panel by X shape with talents (MRX).
- The Finite Element model proposed in this paper shows considerable accuracy for the prediction of maximum shear load and failure mode.
- The developed model has been proved to capture the crack patterns and the stress distribution patterns in both of the brick and mortar.
- The CDP model is an appropriate hypothesis to represent the damage and the non-linear behaviour of brick and mortar.
- The behavior of the masonry is strongly governed by the behavior of the interface, Coulomb friction criterion are important to simulate the load transmission correctly between brick and mortar.

The good correlation between experimental and numerical curves encourages us to use this model in further works for the study of all type of unreinforced or reinforced masonry structure.

In addition, several factors such as type of loading and the characteristics of the single component brick and mortar should be considered in future researches in order to develop a suitable and optimal design approach for strengthening of in-plane loaded masonry walls. The numerical results for strengthened masonry wall will be discussed in future work.

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Evaluation of index and compaction properties of lateritic soils treated with quarry dust based geopolymer cement for subgrade purpose KENNEDY CHIBUZOR ONYELOWE • Department of Civil Engineering, Michael Okpara University of Agriculture, Nigeria • Konyelowe@mouau.edu.ng

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Abstract

The effect of quarry dust based geopolymer cement on the index and compaction characteristics of soft problematic soils has been studied in the laboratory. The particular interest was on the geopolymer cement treatment of test problematic soils subgrade as hydraulically bound materials. In pavement foundation constructions, particular emphasis is always placed on how moisture suction and capillary actions affect the performance of the subgrade layer. Lateritic soils, which form the compacted foundation of pavements have the tendency to behave like plastics or liquids when in contact with moisture. These factors affect substantially the behavior and overtime performance, which is the durability of pavement subgrades. The primary aim of this research work was to adapt geopolymer cement synthesized from valorized solid waste materials (quarry dust and metallurgical slag). Laboratory experiments were used in this study. The test soils were observed to belong to A-2-6, A-2-7 and A-7 soils according to AASHTO classification, which are highly plastic and expansive. They are also of high clay content, a property which makes them unsuitable as moisture bound materials in foundations. The quarry dust based geopolymer cement was utilized at the rate of 10 to 150% by weight of dry soil in an increment of 10%. The test soils treatment protocol shows that the additive consistently improved the plasticity index and maximum dry density of the treated soils obtained at optimum moisture condition. The results have shown that the environmentally friendly cement derived from the valorization of solid waste has improved the properties of the compacted earth utilized under moisture bound environment. So, it stands as a good replacement for ordinary cement.

Keywords: index properties, silicate-based materials, moisture bound composite materials, solid waste valorization, soil compaction, geopolymer cement, geotechnics

Kulcsszavak: szilikát alapú anyagok, szilárd hulladék felhasználása, talaj tömörítés, geopolimer cement, geotechnika

1. Introduction

Index properties of soils are the consistency behavioral patterns soils exhibit within the liquid and plastic phases when they are mixed with moisture. The liquid and plastic behaviors of soft cohesive fine-grained soils determine the soils abilities to be molded, rolled or compacted under different molding moisture conditions [1]. Liquidity of soils is very important in soil mechanics and foundation engineering as it determines certain design factors more especially when the materials are under hydraulically bound conditions [2]. Similarly, the compactibility of soils depend also on the molding moisture conditions or the consistency under which the compaction is carried out [3]. Compaction is a mechanical process of soil stabilization but the state of the soil being compacted plays a big role on the outcome of the compaction process [4]. For instance, highly plastic soils with plasticity index above 17%, are difficult to handle compared to medium, less or nonplastic soils [5]. It is important to note that both properties of consistency and compaction are the responses of soils when mixed with moisture [6]. They both are moisture limits within which soils characteristics are determined [7]. In compaction, the maximum density of soils is achieved at a moisture intake mark called the optimum moisture [8]. The whole aim is to achieve densification of soil mass in a pavement foundation procedure [8]. But in this case, an ecofriendly supplementary cementitious compound has been developed to enhance the index and compaction properties of soft soils utilized as pavement underlain in the laboratory [1-4]. Researchers are working hard in this line to develop composite geomaterials that replace the conventional methods or materials, which bring about greenhouse emissions [9]. And one of such materials is the quarry dust based geopolymer cement, which was utilized to treat soils in varying proportions [10]. This cementitious compound is a combination of quarry dust, metallurgical slag and alkali activators [11]. Both quarry dust and metallurgical slag are industrial waste materials from quarry and metal operations. Quarry dust and metallurgical slag have been in use as stabilization silicate-based agents in the improvement of the mechanical properties of soils especially problematic soils for use as pavement subgrade materials [11]. This has been successful because of the high content of aluminosilicates possessed by these waste materials from guarry and metal operations. Their pozzolanic properties as single independent admixtures have been incorporated into the composite silicatebased cement compound synthesized under the reactive influence of alkali activators [11-13]. This work studied the effect of quarry dust based geopolymer cement on the index and compaction characteristics of treated problematic soils to be utilized as pavement foundation materials.

2. Methodology

2.1 Materials preparation

Three different borrow pit locations, with coordinates 5°29'16" North and 7°28'58" East (Olokoro location), 5°27'0" North and 7°31'60" East (Amaba location), and 5°31'0" North and 7°26'0" East (Ohia location), were the source point of the test soils. Lumps were eliminated by tapping with rubber pestle, and open dried for four days to start of experiment procedure. Fundamental properties, particle size distribution and chemical compounds composition of the three studied soil specimens and test materials are presented in the Table 1, Fig. 1 and Table 2. The test soils have high free swell indexes and low shrinkage limits. This desiccation behavior makes the soils unsuitable as foundation materials. Exposure of these untreated test soils to moisture for a long time creates room for failures of infrastructures constructed on these soils because of the high potential to swell. It is also observed that the soils are highly plastic. This property also makes the untreated material unsuitable to be utilized as subgrade materials or as hydraulically bound materials. The A-2-7, A-2-6 and A-7 test soils are observed to be poorly graded soils [14]. Soils A and C contain

high percentage of clay and are designated as highly clay (CH) content soils, which is a property responsible for the expansivity of clayey soils in contact with moisture. Table 2 presents that the test materials have high percentage of aluminosilicate compounds. Quarry dust (QD) also contains high pozzolanic property [11]. The quarry dust based Geopolymer cement was synthesized according to the procedures and findings of Davidovits [12-13]. The aluminosilicate materials required to materialize the Geopolymer cements consist of quarry dust. The geopolymer cements synthesis was activated by the reactive stimulus of Sodium Hydroxide (NaOH) and Sodium Silicate (Na₂SiO₂) [12-13]. According to previous research findings, a molarity concentration NaOH of 12 was used to achieve an environmentally friendly material handling and construction process and better strength properties of geopolymer cement might be attained. The synthesis of geopolymer matrixes was carried out by mixing these above materials in a proportion of 4.8% activator, plus 80% quarry dust by weight and 15% metallurgical slag by weight [12-13].

Property description of test soils	Values / Descriptions				
and units	Soil (A)	Soil (B)	Soil (C)		
% Passing Sieve, No 200	2.85	10	4.6		
NMC (%)	12.1	13.49	14		
LL (%)	40	46	64		
PL (%)	18	21	36		
PI (%)	22	25	28		
SL (%)	8	8	7		
FSI (%)	250	250	275		
G _s	2.6	2.43	2.12		
AASHTO Classification	A-2-7	A-2-6	A-7		
USCS	GP, CH	GP	GP, CH		
MDD (g/cm ³)	1.76	1.85	1.80		
OMC (%)	13.1	16.2	13.13		
CBR (%)	12	13	8		
Color	Reddish Brown	Reddish Gray	Reddish Ash		

 Table 1
 Basic properties of test soils

 1. táblázat
 A vizsgált talajok alapvető tulajdonságai



Fig. 1 Grain size distribution of studied materials 1. ábra A vizsgált anyagok szemmegoszlása

Materials					Oxide	es compo	sition (co	ntent by	wt %)				
	SiO ₂	Al_2O_3	CaO	Fe ₂ 0 ₃	MgO	K₂0	Na ₂ O	TiO ₂	LOI	P ₂ O ₅	SO₃	IR	Free CaO
Soil A	76.56	15.09	2.30	2.66	0.89	2.10	0.33	0.07	-	-	-	-	-
Soil B	77.57	14.99	3.11	1.78	0.86	1.45	0.23	0.01	-	-	-	-	-
Soil C	77.73	16.65	1.42	3.22	0.07	0.89	0.02	-	-	-	-	-	-
QD	63.48	17.72	5.56	1.77	4.65	2.76	0.01	3.17	0.88	-	-	-	-

*IR is Insoluble Residue, LOI is Loss on Ignition, QD: Quarry Dust

 Table 2
 Chemical oxides compounds composition of the materials used in this paper

 2. táblázat
 A viszgált anyagok kémiai összetétele

2.2 Experimental methods

Preliminary conventional tests; particle size distribution, Atterberg limits, compaction, free swell test and shrinkage limit tests, were conducted to determine the basic properties of the test soils in accordance with British standards [15-17].

Laboratory tests were conducted on the soils treated with varying proportions of quarry dust based geopolymer cement (QDbGPC) at the rate of 10%, 20%, 30% to 150% by weight of the dry soil to the determine the index properties of the treated soils in accordance with British Standards [16]. Similarly, the standard proctor compactive effort was used on test involving moisture/density relationship. Air dried soils samples passing through sieve number 4 (4.76 mm aperture BS sieve) mixed with 10%, 20%, 30% to 150% by weight of the dry soil of the ecofriendly binding admixture were used. The Standard Proctor procedure was conducted in the laboratory in accordance with British Standard [16].

3. Results and analysis

3.1 Consistency limits and compaction behavior

The consistency behavior of QDbGPC treated uncemented lateritic soils are presented in Fig. 2. The plasticity indexes (PI) of the natural untreated lateritic soils were recorded as 22, 25 and 28% respectively. These were considered highly plastic soils and undesirable as construction materials. Upon the treatment of these soils with QDbGPC, the PI was observed to reduce consistently at the rate of 18%, 8% and 4% respectively at 10% by weight utilization of the geopolymer cement. On further treatment with the geopolymer cement at 20% to 100% by weight utilization, the PI consistently reduced at the rate of 9% for treated soils A and B and 13% for treated soil C. At 130%, 140% and 150% utilization of the geopolymer cement, the consistency behavior of soil A showed a steady PI of 4%. This observed behavior of the treated soils has been contributed by increased calcium content from the geopolymer cement blend to hydration reaction and calcination. This was also due to the rearrangement of molecules during the formation of transitional compounds. The hydration and calcination of the treated soils under high proportional aluminosilicate and pozzolanic geopolymer cement had caused this improvement. This however, produced stabilized treated soils of stiff consistency. Moreover, the cations release deposited at the adsorbed complex interface of the treated soils from the synthesized geopolymer cement constituents during the cation exchange reaction had also caused this reaction [18-19]. This

proves that the mechanical factors of the treated soils would remain steady with the usual reduction in the liquid limits on addition of the treatment material if water is used as pore fluid. With the foregoing, under the addition of varying proportions of quarry dust based geopolymer cement, it can be deduced that the liquid limits and plasticity indexes depend on the mechanical conditions rather than the viscosity of pore fluid and density of the treated matrix [18-20]. But to a higher degree, this treatment protocol results is a function of carbonation, cation exchange, polymerization and polycondensation, which are physicochemical properties. Having achieved treated materials matrix of plasticity index below 15%, the utilization of quarry dust based geopolymer cement has improved the treated lateritic soils such that they can best be used at nonfrost-susceptible subgrade and subbase materials. That is to say that under hydraulically bound or moisture bound conditions, the materials can adjust with great flexibility to withstand the effect of that exposure. However, the goal of achieving a durable pavement or foundation facility would have been successful. Similarly, the results of the compaction behavior of the treated lateritic soils are presented in Fig. 3. The maximum dry density (MDD) improved consistently from 1.76, 1.85 and 1.80 g/cm³ respectively at 0% by weight utilization of quarry dust based geopolymer cement to 2.125, 2.5, and 1.95 g/cm³ respectively at 150% by weight utilization of quarry dust based geopolymer cement. On the other hand, the optimum moisture reduced in a consistent trend also. Moisture was required to cause a dissociation of cations and anions from the geopolymer cement during hydration to supply more Ca²⁺ for the cation exchange reaction [11]. This cation exchange reaction caused the consistent reduction of moisture content, which led to the formation of flocs of the clay particles hence the densification of the treated soils.



Fig. 2 Consistency limits of QDbGPC treated soils 2. ábra A QDbGPC-vel kezelt talajok tömörödési határétékei



Fig. 3 Compaction behavior of QDbGPC treated soils 3. ábra A QDbGPC-vel kezelt talajok tömörödési viselkedése

4. Conclusions

The effect of solid waste based composite silicate based geopolymer cement, the quarry dust based geopolymer cement on the treated problematic soils has been observed under laboratory experiments. After the soils were treated through deep mixing with varying proportions of the composite ecofriendly cement, the index and compaction characteristics of the soils showed substantial and consistent improvement. The highly plastic soils improved to less plastic soils with plasticity indexes below 7% while the maximum dry densities obtainable at the optimum moisture condition also improved to good compactness. With the above observations, the quarry dust based composite geopolymer cement has proven to be a good supplementary cementing material for the stabilization of problematic soils and the improvement of the index and compaction properties when used as pavement subgrade materials exposed to moisture conditions.

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Studying compression strength of XD3 concrete samples after addition of calcium nitrate inhibitor and superplasticizers

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Abstract

This research paper presents an analysis of the compression strength properties of concrete samples in the function of their calcium nitrate inhibitor content. This low cost inorganic inhibitor was added to the concrete mix in concentrations of 1% and 3% by weight of cement in addition of two different superplasticizers (MapeiDynamon SR 31 and Oxydtron). The compressive strengths of the so prepared samples were checked according to the relevant European standard and were within the acceptable limits, so this inhibitor does not weaken this important property of the concrete samples. Moreover, the MapeiDynamon SR 31 superplasticizer even showed higher (acceptable) values compared to the other type of superplasticizer due to the difference between their ability in reducing the ratio of water of the concrete samples.

Keywords: calcium nitrate inhibitor, compression strength, superplasticizers, concrete samples Kulcsszavak: kalcium-nitrát uinhibitor, nyomószilárdság, plasztifikáló adalékok, beton minták

1. Introduction

Corrosion of steel rebar in reinforced concrete structures is a serious problem which might cause significant human and economic losses. One of the most important reasons of corrosion in reinforced concrete is chloride attack. The main sources of chlorides are seawater and de-icing salts. Also, there are other sources of chlorides for example from gravels, sand, cement, water (being used for preparing the mixture of fresh concrete) and sometimes from (contaminated) steel rebar. In the subsistence of chloride, the protective passive stratum of steel is locally destroyed and the unprotected steel areas start dissolve. The formation of corroding products (rust) involves a substantial volume increase, i.e. the volume of corrosion products is greater than that of original steel bar. Therefore expansive stresses are induced around corroded steel bars causing possible cracking, spalling of concrete cover and loss of bond between steel and concrete, thus reducing the serviceability of concrete structures [1-9].

There are several methods used to minimize corrosion of steel in concrete, one of these is to use inhibitors. Among the available methods, the uses of corrosion inhibitors are costeffective and easy to handle. Inhibitors are added to fresh concrete while migrating inhibitors are usually proposed for concrete repair. Inhibitors, such as zinc oxide, molybdates and borates, carboxylate ions, quaternary ammonium salts, and other organic compounds were studied [10].

The hardening process of concrete is one of the important factors which effects on the compressive strength of concrete. The binder in concrete is cement. The cement matrix is formed during the hydration process and binds the aggregates together. The basic elements in the production of Portland cement are limestone (CaCO₃), silica (SiO₂), alumina (Al₂O₃), iron oxide

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 (Fe_2O_3) and other substances in minor quantities. To produce cement clinker, the raw materials are mixed, ground and burned in a rotary kiln at about 1450°C. The constituents react to form new minerals. The four major minerals in cement clinker are tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A) and tetracalcium aluminoferrite (C_4AF). The minerals in the clinker begin to react when water is added. This series of chemical reactions is called the hydration process, and consists of various phases. It starts immediately, and the dominant reactions occur on the first day [11, 12]. However, the chemical reactions continue for months, or even years, slowing down gradually. In engineering practice, the hydration process is often supposed to be 'finished' after around 28 days when the representative strength is reached. It is presented schematically in *Fig. 1* according to Locher [13].

In cement hydration process, there are some amounts of calcium hydroxide (CH) remaining in the cement structure. Those remaining CH can be replaced by consuming them with silica to form additional calcium silicate hydrates (C-S-H) which is the main contribution of cement strength [14, 15]. This silicon dioxide (silica) can be found through the pozzolanic material known as pozzolan.

Superplasticizers are preferred in concrete preparation for their ability to reduce the water requirement while maintaining workability and enable the improvement of strength. *Fig.* 2 illustrates the working mechanism of superplasticizer. Cement particles are dispersed by repulsive force generated by negatively charged superplasticizers (*Fig. 2/b*) as, for example, in our case by a modified acrylic polymer Mapei Dynamon SR 31, and the entrapped water would be released. Therefore, the flow characteristics of concrete are improved [16].



- Fig. 1 Scheme of the hydration process according to Locher [13]. (Top) The development of the individual components and (bottom) schematic sketches of the material structure at 4 corresponding stages in time
- ábra Hidraulikus kötés folyamata Locher szerint [13] Az egyes összetevők (fázisok) kialakulása (felül), és a megszilárduló anyag szerkezetének változása vázlatosan az időben (alul)



- Fig. 2 Action of superplasticizer on cement particles. (a) Flocculated cement particles; (b) dispersing cement particles by the repulsive force generated by negatively charged superplasticizer; (c) releasing of entrapped water [16]
- 2. ábra Plasztifikáló adalék hatása a klinker ásványszemekre. (a) Flokkulálódott részecskék; (b) Cement/klinker részecskék diszpergálódása a negatív felületi töltésű plasztifikáló adalék hatására; (c) A fizikailag megkötődött víz felszabadulása [16]

The main purpose of this paper is to present the results of an experimental study on the compressive strengths to monitor and evaluate the effect of calcium nitrate inhibitor and superplasticizers of test samples prepared from concrete type XD3.

2. Materials and methods

2.1 Materials and preparation of samples

Portland slag cement CEM II/A-S 42.5R was used in this study conforming to the standard EN 197-1 [17] and it was received from CRH Magyarország Kft. company in Miskolc, Hungary. Aggregates (fine and coarse) were used according standard EN 12620 [18] and it was also received from the CRH Magyarország Kft. company. This type of cement chosen because it is very workable, has a progressive increase in its initial strength, has very good cement density and its resistance to chemical reactions is high. Calcium nitrate as a inorganic inhibitor and two types of superplasticizers (Mapei Dynamon SR 31 and Oxydtron) were used. Tap water was used for both making and curing the specimens.

Concrete mixes were designed in accordance with the European mix design method (XD3 class) to have compressive strength C35/45 at age of 28 days. Two times three samples were prepared (with the two different plasticizers) and the two sets of three plus three samples contained in 0%, 1%, 3% the calcium nitrate inhibitor as shown in *Table 1*. The concrete cubes so prepared for compressive strength testing had dimensions of 70×70 mm. The molds were thoroughly cleaned and oiled before casting to avoid adhesion with the concrete surface. Mixing of materials was done manually after that water added to the mix with continued mixing, then the mix was put in the molds.

The specimens were taken out from the molds after 24 hours hardening, then were immersed in tap water for 28 day as shown in *Fig. 3*.

Type of Mixtures							
Symbol of Mix	Type of Admixture	Concentration of Calcium Nitrate Inhibitor					
A3 (Reference)	Mapei Dynamon SR 31	without					
B3	Mapei Dynamon SR 31	1% by weight of cement					
C3	Mapei Dynamon SR 31	3% by weight of cement					
A4 (Reference)	Oxydtron	without					
B4	Oxydtron	1% by weight of cement					
C4	Oxydtron	3% by weight of cement					

Table 1The concrete mixtures (specimens) prepared for this study1. táblázatBetonminták összetétele a jelen vizsgálatokhoz



Fig. 3 Curing of compressive strength specimens in tap water 3. ábra Nyomószilárdság mérésére előkészített próbák érlelése csapvízben

2.2 Compressive strength test

The concrete compressive strength was measured by using a compressive strength testing machine (Kispesti Vas és Fém KTSZ) as shown in *Fig.* 4. The cubes were removed from the curing water at age of 28 days after that tested by compressive strength machine as shown in *Fig.* 5. The reported values are the average of three specimens for each age (nine for each mix).



Fig. 4 Machine of compressive strength while sample testing 4. ábra Berendezés a nyomószilárdság mérésére



Fig. 5 Specimens before and after compressive strength testing 5. ábra Betonminták a nyomószilárdság mérése előtt és után

3. Results and disscusion

After adding calcium nitrate as a corrosion inhibitor, the results of compressive strength measurements was presented in *Fig.* 6 and *Table 2*.

Symbol of Mix	Compressive Strength in MPa at 28 day
A3 - Reference (for comparing)	38
B3	41
C3	46
A4 - Reference (for comparing)	30
B4	32
C4	38

 Table 2
 Results of compressive strength test for samples with calcium nitrate inhibitor

 2. táblázat
 Kalcium-nitrát inhibítort is tartalmazó teszt minták nyomószilárdság értékei

As it is illustrated in *Fig. 6* the samples without inhibitor has significantly lower compressive strength. In the samples with Dynamon SR 31 there was not much reduction observed in the compressive strength (about 38 MPa as average for 3 samples tested at age 28 days). While the samples with Oxydtron caused a greater decrease in compressive strength (about 30 MPa as average for 3 samples tested at age 28 days) than Dynamon SR 31 admixture.

(Without this admixtures (Dynamon SR 31 and Oxydtron)

the given type of concrete should have a compression strength of C35/45 MPa at age 28 days.).



Fig. 6 Compressive strength of concrete samples with calcium nitrate inhibitor after immersion in tap water for 28 days

6. ábra Kalcium-nitrán inhibitort is tartalmazó betonminták nyomószilárdság értékei 28 napig csapvízben érlelés után

Comparing the effect of superplastisizers we found that Dynamon SR 31 caused reduction in water during the hardening process of concrete more than Oxydtron and this reduction in water caused increase in compressive strength of concrete and also decreased the porosity as a consequence of a series of chemical reactions (called hydration) of cement with water to form the binding material. The major compounds of cement (tricalcium silicate (C₃S), dicalcium silicate (C₂S), tricalcium aluminate (C₃A) and tetracalcium aluminoferrite (C₄AF)) begin to react with water within a few minutes and absorb the water then do not generate much strength, but can stiffen the mix and reduce workability, so if there's high quantity of water in the concrete that means the hydration components will be larger and these will decrease the capillary porosity and finally cause decrease in strength of concrete because the compressive strength depends also on the capillary porosity. So Dynamon SR 31 superplasticizer in this case more advantageous than Oxydtron superplasticizer.

By using calcium nitrate as corrosion inhibitor in conc. 1% and 3% by weight of cement and Dynamon SR 31 superplasticizer, the compressive strength increased considerably by 8% for 1% addition and 21% for 3% addition, respectively. While after using calcium nitrate inhibitor with 1% and 3% by weight of cement and Oxydtron superplasticizer, the compressive strength effected with an increase 7% for 1% addition and 27% for 3% addition, respectively.

The increase in compressive strength after adding calcium nitrate was due to that this admixture can accelerate the setting time and/or foster the development of higher early strength (calcium nitrate is a multifunctional admixture for concrete, i.e. it can work as setting accelerator for hydration process and as corrosion inhibitor). Calcium nitrate accelerates the hydration process because it has the same Ca²⁺ ion like the cement clinker minerals C₃S and C₂S. And, in presence of the water soluble calcium nitrate additive the crystallization processes can progress more intensively. The observed higher compressive strength with 3% calcium nitrate additive compared to the case with 1% can be attributed to the different compositions of the amorphous calcium silicate hydrate binders (CSH-gels) that will precipitate around the cement grains. An increased calcium

concentration in the pore water when calcium nitrate is also present may stabilize the CSH-gel causing a higher Ca/Si molar ratio and thus leading to the formation of polysilicate anions with shorter average length. The replacement of cement by calcium nitrate on the other hand, will lead to a CSH-gel with a lower Ca/Si ratio and longer length of the polysilicate anions, in addition to the formation of more gel due to the pozzolanic reaction [19-22]. Another contributing factor may be a change in morphology of calcium hydroxide due to a lower solubility caused by a higher Ca²⁺ concentration in the pore water, which agrees with several of researchers [23].

4. Conclusion

After testing the XD3 concrete samples with or without calcium nitrate inhibitors and two types of plasticizers we can conclude the following points:

- The compressive strength without calcium nitrate was higher in samples with Dynamon SR31(A3) than samples with Oxydtron (A4)
- The samples B3 and B4 (with 1% inhibitor) showed increase in compressive strength about 8% and 7% respectively.
- The compressive strength of the sample C3 (with 3% calcium nitrate inhibitor + Dynamon SR 31superplasticizer) was increased by 21%.
- The sample C4 (with 3% calcium nitrate inhibitor + Oxydtron superplasticizer) was the one that increased the most in the compressive strength after the addition of the inhibitor, where the increase was about 27%.

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Material removal characteristics of AI-SiO₂ composite in WEDM

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Abstract

Demand for particle reinforced composite materials gradually increases and that leads to the use of inexpensive particles or solid waste particles as a reinforcement phase to lower the cost of the composite. Silica sand (SiO_2) is an industrial waste that contributes to the strength of the aluminium matrix. Therefore, an attempt is made to use 10 wt. % of silicon dioxide as hardening of particles to obtain composite materials with an aluminium matrix. A stir casting method was used to produce the aluminium matrix. The mechanical and optical microstructural properties of the newly developed Al /SiO₂ composites were studied. Al / SiO₂ composites were processed using wire electrical discharge machining (WEDM) to optimize processing parameters such as pulse-on time (μ s), pulse-off time (μ s) and current (A) to improve material removal rate (MRR). The model was confirmed and good agreement was reached.

Keywords: composite, reinforcement, aluminium, matrix, parameters, current, model, microstructural

Kulcsszavak: kompozit, megerősítés, alumínium, mátrix, paraméterek, áram, modell, mikroszerkezet

1. Introduction

Composite materials, which are usually reinforced with microstructured particles in metal matrices, have the ability to obtain the properties of individual materials. Metal matrix composites (MMC) make it easier for companies involved in the processing of raw materials and the production of goods in factories to meet current and future requirements.Currently, the devotion of time and attention to acquiring knowledge on aluminium (Al) on every side of the planet are largely found owing to the fact that its distinctive property of inferior density, hostility to corrosion and exceptionally good mechanical properties [1]. The manufacturing of part or element from aluminum MMCs make them appealing for many structural component where lofty inflexibility, elevated strength and inferior weight are essential. Aluminum is suited to the weight loss applications [2]. In recent times, the aluminum MMCs have attracted throughout the world attention in virtue of their potentiality to replace massive equivalent, mostly in the automotive and energy area. Toughning of aluminum composite by spreading over the area with exquisitely divided ceramic particles has been evolved for the making of materials with superior stiffness for manufacturing industry. Aluminum MMCs were investigated, and it was established that the hardness of composite materials rises with the inclusion Pondicherry University in recognizing his significant contribution in the area of Thermal aspects in Manufacturing. He has also made remarkable achievement in the areas of teaching, research, invention and extension activities. Starting from a modest career as a Lecturer in a Self-financing College, he rose to the present position of Professor at Pondicherry Engineering College, a premier technical institution. He had guided many dissertations and research projects in Mechanical Engineering and Energy Technology and has 250 Papers to his credit in reputed journals and various conference proceedings. He has teaching and research experience of 30 years out of which 10 years as professor and visited Singapore, Malaysia, UAE and Bangladesh.

of strengthening reinforcements, and superior hardness is by virtue of the existence of tough ceramic matters in the composite [3].

Silica sand is marked as one of the most economical and hard ceramics particles of unambitious density, obtainable in huge amount as a secondary product of solid waste matter in the mass production of a glass factory [4]. The disposal of silica sand powder in a glass factory causes significant economic and environmental problems. The use of quartz sand strengthens the aluminum matrix and offers the advantages of processing industrial waste and reducing processing problems in a glass factory [5]. Therefore, compounds with quartz sand as a reinforcing substance will undoubtedly be beneficial for widespread use in the automotive and aerospace industries. This resarch concentrate on the utilization of unused industrial waste and dispersing it in an aluminum matrix to produce composite by stir casting process [6]. The dissertate for this study is the efficient use of quartz sand, which is one of the residual manufacturing products generated in the glass plant, which argument significant economical and environmental problems. The need for high strength composites enhanced by ceramics particles is also gradually rising. Therefore, more efforts are being made to use silica sand waste being dumped in plants as a reinforcement particles to lower the price of the cost of the composite materials.

In the present work, the lack of hardness of pure Aluminum is amplified by incorporating 10 wt. % SiO₂. Wire EDM Process (WEDM) is a potential process that is useful for processing hardto-manage materials [7]. WEDM is used to process advanced materials with a metal matrix (MMC) and conductive ceramics. Efforts were made to treat freshly prepared Al composite with 10 wt. % SiO₂ back up to hold superior material removal rate (MRR). Measured WEDM coefficients, such as pulse time (μ s), pulse turn-off time (μ s), and current (A), have been optimized to refine the MRR. Analysis of variance (ANOVA) was put to use as a means of finding the major noteworthy measurable factors in WEDM operation . Taguchi's L9 orthogonal matrices were over-decorated to estimate the net parameter space in just a few experiments.

2. Materials and manufacturing

Pure commercially available Aluminum is taken as a matrix, and 10% via weight of silica is taken as reinforcement. The properties of SiO_2 are proven in *Table 1*. The common particle size of SiO_2 used on this work was 50 µm.Although many production generation had been used for the producing of mining and metallurgical composites, stir casting technology were one of the dominant technology used within the industry. This method is commonly used to produce complex types of materials for the manufacturing industry.

Chemical Properties						
Crystal name Monoclinic						
Mineral name	Moganite					
Chemical Formula	SiO ₂					
Mesh size	230					
Size	53 µm					
Thermal Properties						
Melting point	1760°C					
Boiling Point ^o C	2240°C					
Physical Properties						
Density g/cm ³	2.33					
Molar mass	59.96 g/mol					

Table 1 Properties of silica sand

1. táblázat Kvarchomok tulajdonságai

In addition, the constant propagation of reinforcement in the matrix is very feasible due to constant agitation by the mixer. In this study, to achieve all of these benefits, a method of stir casting method was selected for the manufacture of MMC [8,9]. The required amounts of silica are stored together with a graphite crucible, motor and stirrer. Al-SiO₂ compounds were synthesized using a stirrer.

Pure Aluminum is melted in a coal furnace. A vortex was created at a temperature of 770 °C and then preheated quartz sand was gradually added to the molten metal and then thoroughly mixed with a stirrer for 20 minutes [10]. Uniform dispersion of the reinforcement in the matrix is achieved using a stirrer [11]. The drive speed was 550 rpm. *Fig. 1* shows die casting of a matrix consisting of Aluminum-SiO₂.



Fig. 1 The casting of Al/SiO₂ 1. ábra Al/SiO₂rudak

3. Results and discussion

Since the cutting operation can change the microstructure of the sample due to heating, cutting was done using a lowvelocity diamond plate [12]. After assembly, grinding was performed to reduce surface damage caused by cutting discs. The grinding speed was maintained at about 150 rpm and was performed with increasing paper fineness of 240, 400, 600 and 1000. Small scratches caused by the final grinding operation are removed by polishing using powder flakes. Fig. 2 shows a uniform distribution of reinforcement throughout the matrix. The particle content is smaller since the area is smaller. With a greater growth in the quantity of composite particles, the grain obstacles are visible. Reinforcing particles are dark in colour. The distribution of quartz sand is seen evenly in the Aluminum matrix. This is achieved by vigorous stirring with a stirrer. There are no pores around the quartz sand particles. Besides, there is no matrix region without reinforcement. It has also been found that there is a good interfacial compound in addition to a uniform distribution. The tensile strength (Fig. 3) of 0% by mass of Al is 7 MPa, and this value increases to 20 MPa for Al / SiO, with the addition of 5% by mass of SiO₂, which shows an improvement of 185% compared to pure Al. Similarly, for 10 wt. % SiO₂, the tensile strength will increase to 29 MPa, that is approximately 315% better than the tensile power of unreinforced Aluminum. This is because the reinforcing particles in the compounds of the metal matrix are evenly distributed. But adding more reinforcing particles leads to agglomeration, which reduces the bond strength between Al and the reinforcing particles. An increase in SiO₂ will lead to greater cracking and consequently to lower tensile strength. Besides, contacts between silica particles will isolate ductile Aluminum.



Fig. 2 Microstructure of AMC 2. ábra AMC mikroszerkezete



3. ábra Húzószilárdság SiO, tartalom függvényében

As a result of this effect on hardness, it was determined that the value of hardness is better with increased traction. It is also noted that the maximum hardness is attained at Al with 10 wt% SiO₂. The hardness cost of pure Al turned into 26 BHN, which became elevated to 30 BHN with the addition of five wt.% SiO₂ within the Al matrix and 33 BHN for 10 wt.% SiO₂.Improvement of hardness value is achieved for 5 wt% SiO₂ is 15% and similarly, for 10 wt%, SiO₂ is 26%. Increase of the hardness value shows that the addition of silica particles increased to 10%, the hardness increased. This is because the harder SiO₂ particles replace some of the soft Aluminum. However, when the addition of reinforcement increased beyond 10 wt%, the hardness decreased. This is because more silica particle presents in the Al matrix then the bonding becomes poor. Hence beyond the addition of 10 wt% SiO into Al matrix lead to low hardness. Fig. 4 demonstrates the hardness estimations of the ALMMC. Uniform distribution of SiO₂ into Aluminum matrix would increase hardness and also ductile Aluminum would become a little bit brittle.



Fig. 4 Hardness vs wt % of SiO₂ 4. ábra Keménység SiO₂ tartalom függvényében

The results confirms that 5 wt.% SiO₂ with aluminium composites gives the yield strength of 22 N/mm² which is 100% higher than yield strength of pure aluminium (11 N/ mm²). Similarly, for 10 wt.% SiO₂, the yield strength is 28 N/ mm², which gives an improvement of 154% compared to unreinforced Al. Fig. 5 shows the yield strength of the metal matrix composites. The uniform dissemination of SiO₂ in the aluminum lattice essentially improved the yield quality. Fig. 6 shows that at 5% by mass of SiO₂, a decrease of 5%, 5% by SiO₂ and 3.7% at 10% by mass of SiO₂ is observed, while at 5% by mass of SiO₂ and 10% by mass, a decrease in SiO₂ is achieved using unreinforced Aluminum reaches 80%. Because of warm confound, the Al grid enters the stress state, and SiO₂ particles enter the compression state. Since SiO₂ has low elasticity and a high decrease in compressive quality is accomplished. It was noted that the newly created unaltered aluminum was strengthened by 10 wt. % SiO₂ has high resistance. Since it is very durable, conventional machining may not be suitable for machining operations. Therefore, the experiments were carried out in wire-cut EDM (EDM DK7740), which is shown in Fig. 7. Specifications of WEDM machine are given in Table 2. Molybdenum wire (DK7740) is used as the electrode wire in the EDM for wire cutting. To ensure a smooth movement of the wire feed, digital circuits controlled by an inverter are used. This molybdenum wire can be used for quite a long time with high accuracy and can reduce wire breakage. The width of the molybdenum wire used in this machining was 0.18 mm. EDM molybdenum cutting machines are mainly used in electrical products, automotive components, military industry, etc. Water is utilized to control resistivity and other electrical properties. Due to the high-temperature discharge, the dielectric cleaning fluid removes the surface that melts and the particles that are cut [13]. The research sequence was carried out on WEDM machine [14]. The selected criteria for the preparation of productivity were MRR and it can very well be represented (equation 1) as the ratio of the difference in weight during handling to the time and thickness of the composite [15]. Readings were recorded using the extended scale.



Wt% reinforcement

Fig. 6 Elongation vs wt % of SiO₂
6. ábra Megnyúlás SiO₂ tartalom függvényében



Fig. 7 Wire cut EDM machine 7. ábra Huzalos szikraforgácsoló gép

S.No	Item	Unit	DK7740
1	Travel of X/Y	mm	400 x 500
2	Table Size	mm	500 x 785
4	Maximum load of table	kg	500
5	Dimension (LxWxH)	mm	1700 x 1350 x 1700
6	Weight of machine	kg	2000
7	Best Finish	Ra≤µm	≤ 1.5

Table 2 Specifications of WEDM

2. táblázat WEDM eljárás műszaki adatai

 $MRR = (W_{ij} - W_{ij}) / (\rho_{composite} * t)$ (1) where $W_{ij} \& W_{ij}$ are the weights, t is the machining time.

To plan the experiments, Taguchi L9 orthogonal matrices were used. The Taguchi strategy is a procedure of value improvement, and this technique is utilized to lessen contrasts in item configuration and to improve quality. This strategy is known for its quality optimization [16]. Based on the trial configuration, tests were led, and the size of the chose piece was 10 mm. The test ought to be completed by changing the procedure parameters, which regularly influences the preparing procedure to get the required quality characteristics [17]. In this investigation, the Taguchi strategy is utilized to create orthogonal array (OA) for a sum of 3 parameters, namely pulse-on time, pulse-off time and current as shown in Table 3. The investigation expects to get a higher pace of material evacuation.In the present work, Analysis of Variance (ANOVA) was performed for a combined response variable using Minitab-19, statistical software and significant parameters responsible for the maximum MRR were listed in Table 4. It was found that the current has a significant effect (60.1%) on the MRR, followed by the pulse-off time (13%) and the pulse-on time (12%). The response data of the factor are presented in graphical form in Fig. 8. The best possible processing parameters are A1, B1 and C3. Table 5 and 6 shows the reaction and ANOVA results respectively. By establishing the identified optimal values of the control parameters as input parameters (A1, B1 and C3), a control experiment is carried out using the same experimental setup and is repeated twice [18,19]. The values of the qualitative characteristics obtained during the verification experiments have an MRR of 16.1. The results of the validation tests are appeared in Table 7.



Fig. 8 Main effects plots for S/N ratios

8. ábra S/N arányok főbb hatásai

Parameters	Level-1	Level-2	Level-3
Pulse on time (µs)	6	7	8
Pulse off time (µs)	5	6	7
Current (A)	1	2	3

Table 3 Process parameters and their levels

3. táblázat Az eljárás paraméterei különböző szinteken

	Pulse on Time (µs)	Pulse off Time (µs)	Current (A)	MRR
1	6	5	1	13.9
2	6	6	2	9.99
3	6	7	3	15.2
4	7	5	2	10.2
5	7	6	3	15.3
6	7	7	1	7.13
7	8	5	3	15.3
8	8	6	1	13.2
9	8	7	2	10.1

 Table 4
 Experimental layout using L9 OA and performance result

 4. táblázat
 Kísérleti elrendezések L9 OA felhasználása esetén és eredményeik

Level	A	В	С
1	22.16	22.24	20.78
2	20.31	22.03	20.08
3	22.06	20.26	23.67
Delta	1.85	1.98	3.59
Rank	3	2	1

Table 5Response table for signal to noise ratios5. táblázatJel-zaj arányok válaszreakciós táblázata

Source	DF	Adj SS	Adj MS	F-Value	P-Value	%contribution
Pulse on time (µs)	2	8.624	4.312	0.90	0.526	12
Pulse off time (µs)	2	9.570	4.785	1.00	0.500	13
Current (A)	2	43.328	21.664	4.53	0.181	60.1
Error	2	9.557	4.779			
Total	8	71.079				

Table 6 Analysis of variance (ANOVA)

6. táblázat Varianciaanalízis (ANOVA)

C No Itomo		Initial cutting	Optimal machining parameters	
3.110	items	parameters	Prediction	Experiment
1	Level	A1B1C1	A1B1C3	A2B2C1
2	MRR	13.9	16.9	16.1
3	S/N ratio	22.8603	25.0557	24.1365

Table 7 Results of the confirmation test

7. táblázat Teszteredmények

4. Conclusion

Quartz sand waste discharged into the open area was collected and successfully used as reinforcement in a pure Aluminum matrix. In this study, a stir casting method was chosen for the manufacture of MMCs, as this is the cheapest manufacturing method used to make complex moulds. The mechanical properties of the newly manufactured Al / SiO, compounds improved significantly until the reinforcement level reached Al + 10% SiO₂. The hardness of pure Al was 26 BHN, increased to 30 BHN with the addition of 5% by weight of SiO₂ to the Al matrix and 33 BHN for 10% by weight of SiO₂. A rise in the hardness of 5 wt. % SiO, is 15%, and similarly for 10 wt. % SiO₂ is 26%. It was shown that the elastic limit of 5% by weight of SiO, showed an elastic limit of 100% higher. Similarly, for 10 wt. % SiO₂, the elastic limit is 28 N/ mm², which provides an improvement of 154% compared with unreinforced Aluminum. The range of recovery results showed that a decrease of 4% for 5% by weight of SiO₂ and by 3.7% for 10% by weight of SiO₂.79% for a decrease of 5% by weight of SiO₂ and 80% reduction for 10% mass of SiO₂ with unreinforced Al.An optical microscope image shows that SiO₂ particles are well distributed in the Al matrix. It was concluded that Al with 10 wt. % SiO₂ can be considered as a suitable material in sectors where lightweight with improved mechanical properties is required. Therefore, Al + 10% by weight of SiO₂ obtained by casting with stirring are taken for Wire EDM cutting experiments. The current, which is the most significant parameter of 60.1%, gives a vital effect with a subsequent pulse-off time of 13% and the inclusion of a pulse on time of 12%. The percentage of errors (4.94) is in the acceptable range, which is less than 5%.

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Superficial hardening improvement of nano and micro composite AI TiC

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Abstract

The present investigation aims to study the improvement of the composite surface hardness by air abrasive jet polishing (AJP) of SiO₂ particles. The experiments were conducted by synthesizing Al-TiC composites through melting the aluminum at 850°C and added TiC gradually for 10 minutes to the liquefied aluminum with different sizes and ratios (300-500 nm and 100-200 µm) and (5, 10, 15, 20 and 25 wt.%) respectively of TiC. The molten Al-TiC was poured to a previously prepared sand mould. The hardness improvement for pure Aluminum, Al-micro-TiC and Al- nano-TiC composites were 7%, 11%, and 15% respectively. The obtained results demonstrate the importance of superficial hardening of composites Al-TiC by impact of the SiO₂ air jet and show that the surface hardness improvement is greatest for the nanocomposites compared with micro-composite and pure matrix material.

Keywords: abrasive jet polishing, nano and micro composites Al-TiC, hardness improvement, SiO₂ Kulcsszavak: csiszolófúvós polírozás, Al-TiC nano és mikro kompozitok, keménység növel, SiO₂

1. Introduction

The surface finish of Al-TiC components plays an important role in the mechanical response and wear resistance. Therefore, the surface treatment is a primordial process to achieve certain qualities that are not available from the primary manufacturing processes. One of the effective surface treatment processes is the polishing process using abrasive jet of particles. The effect of polishing techniques and penning on surface roughness and hardness are widely studies on composites [1]. Abrasive jet machining (AJM) is a process that does not include a physical contact between the tool and work piece so there are no thermal stresses and shocks developed. AJM can be used for many processes such as cutting, cleaning, polishing, etching, drilling and finishing operations. The effects of polishing techniques on surface roughness and micro hardness of resin composites are desirable in order to reduce the number of clinic sessions and bringing more comfort and satisfaction to the patient in dental application [2-4]. In addition, the air abrasion is commonly used on surface treatment for porcelain veneers [5]. The effect of different air-abrasion particles on metal-ceramic bond strength is widely analyzed in the work of Tolga Kulunk et al. [6]. Other studies using abrasive water jet polishing were conducted to improve the fatigue strength of metals [7, 8]. F. Boud et al. [9] studied the impact of plain water jet machining on the surface integrity of aluminum 7475. Others focused on penning treatment such as Kyun-Taek Cho et al. [10] who worked on surface hardening of aluminum alloy by soft-shot polishing treatment with Zn based ball. In this work, a nano and micro composites of Al-TiC are chosen as testing materials. Several previous works have been done in the synthesis of Al-TiC composites [11, 12]. In an attempt to improve the Al-TiC composites surfaces hardness with different TiC sizes and ratios a high efficient and low-cost AJP machine is designed to conduct the proposed testing.

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2. Experimental procedures

Aluminum (Al) was used as matrix material whereas TiC particles with different sizes and ratios (300-500 nm and 100-200 µm), (5, 10, 15, 20 and 25wt. %) were used as reinforcement in the Al-TiC composites. Melting of Al was carried out using a resistance furnace operating at 850°C. The reinforcements were then added gradually for 10 minutes to the molten aluminum with different weight ratios (5, 10, 15, 20 and 25) wt.% of TiC. Mixtures were stirred for 10 minutes after the additions of TiC. The stirrer rod was operated at 450 rpm to ensure a good distribution and homogeneity of the nano and micro TiC powder in the molten Al matrix. With 10 °C/min heating rate the furnace, the temperature was held at 850°C to ensure high fluidity of Al. After the Al-TiC became homogenous, the liquefied Al-TiC was poured in to previously prepared sand mould. However, at a temperature of 700°C the molten became in a semi-solid state, where the slurry cannot be poured into the mold because at this stage, its viscosity is high and the fluidity is greatly low. Therefore, before pouring the slurry into the mold, it was necessary to re-melt it to a molten condition, and re-stir before pouring [13]. The abrasive jet machine (AJM) is utilized in the unconventional machining process of material removal from a work piece by the application of a high speed stream of abrasive particles carried in the air medium from a nozzle as shown in Fig. 1.

The AJM is used to cut hard and brittle materials where a three-axis CNC machine is utilized for material removal process. The machines are designed and selected through appropriately to o the tasks. To design and construct the AJM components several factors have to be considered: compressible flow law, carrier gas, flow rate, vibration level, nozzle size, stand-off effects, material removable rate estimation, etc. The selection of parts, the assembly process, the fabrication and the test of the machine performance were carried out. The AJM was designed and constructed to perform a CNC engraving, cutting and drilling as shown in *Fig. 2*.



Fig. 1 Schematic representation of the AJM 1. ábra AJM sematikus ábrázolása





Fig. 2 The abrasive jet machine (AJM) (a), the nozzle (b) 2. ábra AJM (a), fúvóka (b)

The AJP was designed with abrasive Silica sand (SiO_2) and used with a 90° impact angle and average gas pressure of around 5 bar with 2 mm nozzle diameter and a standoff height of 10 mm. The mass flow rate of SiO₂ was set to 1.2 gram/s. Total polishing time was 10 seconds. Control of the nozzle position and the blasting time on the top surface were controlled by a CNC table. Hardness test evaluation was carried out through the Brinell test machine WP 300 from GANT Company. Brinell hardness test carried out at 9.8 KN for 30 seconds. The test was used to investigate the influence of sizes and weight fraction of TiC on the matrix hardness. A total of five samples were taken for each ratio of the Al-TiC to obtain the average hardness value for an accurate result. The concept of hardness improvement by abrasive jet polishing of SiO_2 particles is represented in *Fig. 3.* The Silica sand SiO_2 particles are characterized by a large number of irregular angular surface features and have a relatively high hardness (Mohs Hardness 6-7).



Fig. 3 The concept of hardness improvement by abrasive jet polishing of SiO₂ 3. ábra A keménység növelésének koncepciója SiO₂ csiszolófúvós polírozásával

In order to evaluate the efficiency of the abrasive polishing machine, a primary experiment was carried out on four known specimens delivered from GANT Company under standard references as shown in *Table 1*. The polishing test was performed using aluminum alloy EN AW-6082 (called in ISO: Al Si1MgMn), copper CW004A (designation for the 99.9% pure copper), Brass CW614N with 37-45% Zinc, steel alloy S235 – IAC-C with maxi C% of 0.22 and Mn % of 1.6.

Based to the experimental results grouped in *Table 1*, it is observed that the increasing of the hardness of the aluminum alloy is about 51%, for the copper alloy is 73%, for brass is 53% and for steel is 29%. The improvement of surface hardness was significant, which confirm the effectiveness of the present jet polishing process on the material surface finishing.

Material	Brinell Hardness before polishing	Brinell Hardness after polishing t*=10s d*= 10 mm Polishing at MP=1.2 gram/s	Percentage of hardness improvement MP*=1.2 gram/s
Aluminum Alloy EN AW-6082	67 HB	101 HB	51%
Copper Alloy CW004A	55 HB	95 HB	73%
Brass CW614N	90 HB	138 HB	53%
Steel S235-IAC-C	130 HB	168 HB	29%

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**t(s): Time of polishing.*

*d (mm): Standoff distance between nozzle and top surface of the workpiece. *MP (gram/s): Mass flow rate of abrasive particle of SiO,

 Table 1
 Hardness improvement of standard specimens by polishing test

 1. táblázat
 A minták keménységének növekedése polírozási teszt alapján

3. Results and discussion

3.1 Hardness results before polishing

In this part, hardness test was conducted to investigate the influence of sizes and weight fraction of TiC on the matrix hardness. A total of five samples were taken for each ratio of Al-TiC to obtain the average hardness value for an accurate result. The hardness values of the micro and nano composites depend on the amount of particles, sizes, and uniformity distribution of TiC particles in the Al matrix as shown in *Fig. 4*.



Fig. 4 Comparison between average HB for nano and micro 4. ábra Átlagos Brinell-keménység összehasonlítása nano- és mikro kompozitokon

It is observed from the results of hardness in *Fig. 4* that all reinforced specimens have a higher hardness than that of the matrix material. In general, the hardness increases with increasing the hard particles (TiC). The hardness for specimens with 20 wt% and 25 wt% TiC decreases due to the agglomeration of TiC particles. For these samples, the highest hardness measured is at 15 wt% Nano TiC at 28 HB while the minimum hardness is at 0 wt%, and that proves that the aluminum reinforced with TiC improves the hardness. Moreover, it was observed based on *Fig. 4* that the nano-particles of TiC gives a little higher hardness for the composite Al-TiC compared with micro particles. The decrease of hardness at 20 wt% and 25 wt% of TiC is due to the effects of grain boundary.

The presented results had been observed in several investigations, which indicated the decrease of hardness below a critical grain size [17], [18] and [19]. Both experiments and simulations in [20], [21] and [22] have also shown that the strength/hardness decreases with further grain refinement below the critical value. This indicates the occurrence of a shift in the dominated deformation mechanisms from dislocationmediated plasticity to grain-boundary-associated plasticity such as grain-boundary sliding, grain-boundary diffusion and grain rotation. Due to the small size of powder (especially in nano powder) a change is evident in hardness value for each specimen. If a particle is dispersed in the composite, a better value for hardness would be obtained. The agglomeration that occurs affects the mechanical behavior of the specimen. This is due to the powder collected at one portion of the specimen that causes a soft and porous surface.

3.2 Hardness improvement after polishing of the composite Al-TiC

All specimens were tested for Brinell hardness before the polishing. The outer surfaces of the workpieces were machined using an abrasive jet at a standoff of 10 mm and mass flow rate of abrasive particles of 1.2 gram/s. Then, the Brinell hardness test was repeated again on the same specimens. The degree of surface roughness was changed and the hardness as well. The amelioration of the hardness of the nano and micro composite Al-TiC simples at different ratios of TiC are regrouped in *Table 2*.

The percentage rates of hardness improvement in specimens were calculated through five experimental tests on each specimen. The average is presented in *Table 2* for microcomposite and nano-composite with different ratios of TiC. By comparing the hardness before and after polishing it is clearly noticed that the increasing of hardness is about 7% to 15% for nanocomposites and about 6% to 11% for micro-composites due to the impact

of SiO_2 particles by abrasive jet polishing. This process produces tensile stresses in the surface because the surface is trying to become plastically larger.

As a reaction to the polishing or peening treatment a compressive unused stress is formed in the surface layer. These compressive unused stresses have to be balanced by unused tensile stresses in the entirely elastically deformed component interior.

Material specimen micro composites	Before polishing (HB)	After polishing (HB)	Percentage of hardness improvement
Pure Al	26.2	28	7%
Al- 5wt% TiC	26.7	28.7	7%
Al- 10wt% TiC	27.3	29	6%
Al- 15wt% TiC	27.7	30.7	11%
Al-20wt% TiC	26.9	29	8%
Al- 25wt% TiC	26.3	28.5	8%
Material			

Materiai specimen nano composites	Before polishing (HB)	After polishing (HB)	Percentage of hardness improvement
Pure Al	26.2	28.4	8%
Al- 5wt% TiC	26.9	28.8	7%
Al- 10wt% TiC	27.6	30.5	11%
Al- 15wt% TiC	28	32.3	15%
Al-20wt% TiC	27.4	31	13%
Al- 25wt% TiC	26.7	29	9%

 Table 2
 Hardness improvement for micro, and nano polishing Al- TiC composites

 2. táblázat
 Az nano- és mikro polírozott Al-TiC kompozitok keménységének növekedése

The position of the maximum of compressive residual stresses depends on several parameters such as: the compressible flow law, carrier gas, flow rate, vibration level, nozzle size, stand-off effects, material removable rate estimation, etc. With increasing the shot peening time, deeper surface layers are plastically deformed, until a saturation is reached, which depends on the energy of the impact shot. The surface shows an increased dislocation density and roughness after shot polishing as in *Figs. 5(b)* and 6(b). This increase in the dislocation density leads to an increase in the material strength of the surface. This hardening of the polishing Al-TiC composite surfaces have been easily proven by Brinell hardness as shown in *Table* 2. In addition, it is noticed that the hardness decreases after reaching 15 wt% of TiC in the aluminum matrix due to the high content of TiC in the Al matrix, which is very sensitive to the agglomeration of TiC.

3.3. Microstructure Image Analyses

The micrographs shown in *Figs.* 5(a) and 6(a) clearly display the distribution of the TiC particles (dark areas) in the Al matrix (bright areas). As shown in *Figs.* 5(a) and 6(a), the small particles are distributed homogenously among the big particles (agglomeration of many small particles together). It should be noted that the particle sizes of the small particles are uniformly distributed. However, some pores can be observed. The formation of pores is mainly due to the non-uniformity of the initial TiC particles. Moreover, it is clear from *Figs.* 5(a)and 6(a) that the reinforcement particles of the composites are embedded in the aluminum matrix. A small agglomeration of TiC particles in the aluminum matrix has been noticed, and this is mainly due to the non-homogeneity involved in the mixing process carried out before casting. The size of the TiC particles in the composites increases as the TiC content increases.







(a) (b)
 Fig. 6 Microscopic observation of the microcomposite Al TiC at Wt 10% before polishing and (b) after polishing
 6. ábra Al TiC mikrokompozitok mikroszkópos vizsgálata

 (a) polírozás előtt és (b) polírozás után

The surface shows an increased dislocation density and roughness after shot polishing as in *Figs.* 5(b) and 6(b). This increase in dislocation density leads to an increase in the porosity and material strength in the surface. The increase in roughness inherited during shot peening must always be considered as negative. At higher magnification overlaps, micro-cracks and areas with an obvious turbulent flow can be found. With the increase in peening time the surface roughness increases as well. A compressive residual stresses can be done to drastically reduce the faster crack propagation rate of the homogeneously deforming surface zone.

4. Conclusion

The presented study exposes the improvement of hardness for composites surfaces by Abrasive Jet Polishing (AJP). Composites of Al-TiC have been synthesized successfully by stir casting process with different sizes and ratios of TiC (300-500 nm, and 100-200 µm) and (5, 10, 15, 20 and 25 wt.%) respectively. An AJP by abrasive silica sand (SiO₂) was used to conduct the experiments. The increases in surface hardness for aluminum alloy, copper alloy, brass, steel were about 51%, 73%, 53%, and 29%, respectively. Hardness improvement for pure aluminum, Al-microTiC, Al-nanoTiC composites were 7%, 11%, and 15%, respectively. From the introduced results, improvements in polished surface workpiece are limited by the SiO₂ grainimpact phenomenon and the embedment of SiO₂ fragments in the workpiece surface. The surface shows an increased dislocation density and roughness after shot polishing. This increase in dislocation density leads to an increased porosity, material strength in the surface, and reduction the faster crack propagation rate of the homogeneously deforming surface zone. The obtained results on Al-TiC composite demonstrate the importance of superficial hardening of composites by impact of the SiO₂ air jet and show that the surface hardness improvement is greatest for the nanocomposites compared with micro-composite and pure matrix material.

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Creation of "necessary" mixtures of baking soda, hydrogen peroxide and warm water as a strategy for modernization bleaching cleaners of ceramic

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Abstract

Hydrogen peroxide solutions have been used for more than 100 years as industrial bleach for various wood, paper, textile, leather products, as well as for hair whitening. Since the same time, hydrogen peroxide solutions and sodium bicarbonate solutions have been widely used in medicine as antiseptic and disinfectants to remove pus from purulent wounds. Less than this period, hydrogen peroxide solutions are successfully used for bleaching fish and various seafood. However, until the early 21st century, the combination of hydrogen peroxide with sodium bicarbonate was not used as a bleaching cleaner for surfaces contaminated with blood and/or pus stains. The use of hydrogen peroxide solution in combination with sodium bicarbonate as a universal bleaching and dissolving medical cleaner was first proposed in the last 20 years in Russia. It was during these years that a new group of drugs was discovered in pharmacology: drugs, bleaching and dissolving blood stains and a mass of thick pus. It is proved that the local application of various mixtures of baking soda, hydrogen peroxide and warm water provides a safe urgent dissolution and discoloration of the dense biological masses. It is established that purification, bleaching and removal of organic pollutants is achieved due to their hyperthermal softening, cavitation loosening, alkaline saponification and dissolution, as well as due to oxidative discoloration of pigments. It is proposed to use the created Arsenal of biomedical alkaline bleaching cleaners for the analysis and development of new effective and safe ceramic bleaches in everyday life.

Keywords: teeth, dentures, ceramic, hydrogen peroxide, sodium hydrogen carbonate, bleaching agent

Kulcsszavak: fogak, fogsorok, kerámia, hidrogén-peroxid, nátrium-hidrogén-karbonát, fehérítő

1. Introduction

One of the distinguishing features of high-quality ceramic tableware and many other ceramic, bioceramic, clay and metal products intended for storage, preparation and consumption of food products is the presence of enamel on their working surface. This enamel in its physical and chemical properties is very similar to the enamel that covers the working surfaces of mollusk shells, human and animal teeth. Such enamel is also covered of modern ceramic and/or metal-ceramic dentures and braces [1]. The common property of all these enamels is that they have a very high strength and a very smooth and slippery surface that does not even have a micropore. Therefore, the surface of these enamels has low adhesion to organic substances and microorganisms and is easily cleaned from dirt when washing [2]. In turn, the purity of the enamel surface affects the contamination of products and determines the aesthetic properties of the product [3].

In addition, the cleanliness of the working surfaces of the discussed products is the main task of sanitary and hygienic control of their quality. The fact is that the remains of food products, stuck to the surface of the enamel, can turn into a kind of fertile bed for the reproduction of microorganisms, of which some part can become a breeding ground for infectious diseases. Therefore, in practice, the contamination of surfaces is investigated not so much for the presence of food products on it, as for their contamination with microorganisms [4,5].

Therefore, it is no accident that wherever objects and products covered with enamel are used, hygienic means are constantly and repeatedly used to care for them, or rather-to clean the enamel surface from mechanical and microbiological contaminants. Therefore, at all times the production of ceramics and enamels has been inextricably linked with the production of disinfectants, detergents, bleaching and hygiene products [6].

In the second half of the 20th century, the world has increased interest in household ceramic products. In the late 20th century and early 21st century, the demand for ceramic and bioceramic medical products increased. Nowadays, the demand for ceramic products used in dentistry is especially great [7].

Due to the high demand for ceramic products, their quantity, range and quality has increased significantly. At the same time, the use of known detergents and devices (washcloths, sponges, wipes, mops, brushes, Soaps, shampoos, washing powders, washing machines, food processors, etc.), personal

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is Professor, Leading Specialist in Udmurt Federal Research Center, Head of Department Pharmacology in Izhevsk State Medical Academy, author of 220 inventions. Honored inventor of the Russian Federation hygiene products in the oral cavity (toothpastes, toothbrushes, toothpicks, elixirs, etc.) still does not allow you to quickly achieve whiteness and purity of the surfaces of enamels covering cookware, baths, sinks, ceramic tiles, as well as teeth and dental constructions [1,8]. Therefore, within a few days or weeks after the start of the use of ceramic products, their surface becomes dirty and remains dirty regardless of the quality of enamel and hygiene products. It impairs the appearance of household products (such as cups, dishes, pots, pans, knives), teeth, dentures, braces and causes bad breath in humans and animals [9]. All this encourages researchers to search for new more effective means of washing and whitening enamel covering ceramic products.

The analysis of known detergents and cleaning agents, devices and methods of their application was carried out. It is shown that most traditional technical solutions are based on the principle of mechanical cleaning of the enamel surface from food contamination [10]. However, the effectiveness and safety of known detergents and bleaching agents and methods of their application is still not completely satisfied with consumers. At the same time, progress in this direction has slowed. This is probably due to the fact that the potential of this principle is almost completely exhausted. That is why to optimize the search for new tools, it is proposed to pay attention to a different principle of washing and whitening enamels.

In particular, as a new principle of enamel cleaning, the principle of physical-chemical dissolution of adhesives is proposed, which bind together all the residues of organic substances into one dense biomass, visible to the eye and assessed as a stain of pollution. It is shown that this principle can be realized with the help of hydrogen peroxide solution, to which sodium bicarbonate and/or oxygen gas under excessive pressure are added in excess [11]. Such a solution provides urgent purification and bleaching of the enamel surface from biological pollutants due to alkaline saponification of protein and protein-lipid complexes, "explosive" loosening of biomass due to cold boiling caused by the release of gas (in particular, oxygen) inside this biomass, and oxidative destruction of lightabsorbing double bonds in pigments [12-14]. In this case, such a cleaner is safe for the consumer, as it includes edible ingredients [15].

It is demonstrated that this principle is successfully implemented in medicine for urgent dissolution of thick purulent masses, sulfur plugs and dried blood [11]. For this purpose, special technologies for the local application of alkaline aqueous solutions of hydrogen peroxide heated to 37 – 42 °C, saturated with oxygen gas under increased pressure and sometimes supplemented with an insoluble material with frictional properties are proposed [16]. It is noted that these physical and chemical factors have long been considered very important in terms of physical and chemical sciences. However, further research into their use in the field of hygiene is needed in order to develop more effective and safe cleaning and bleaching agents suitable and safe for the treatment of ceramic products in the home.

At the same time, the influence of these and other physical and chemical factors on the removal of dirt from the surface of enamels of ceramic products is still insufficiently studied.

2. Materials and methods

Since 1999, the search for patents for inventions and scientific information on new means and methods of dissolution and removal of dense biological masses using physical and chemical factors of local interaction has been continuously conducted. The search strategy keywords were as follows: brushes, mops, brooms, washcloths, soaps, shampoos, laundry detergent, washing powder, bleach, cleaner, tooth cleaner, thick pus solvent, toothpaste, blood stain, pus, tattoo, plaque, tooth, denture, ceramic ware, textiles, ceramics, skin and bruise. Key words were limited to means and ways of using them for hygienic and aesthetic purposes.

The criteria for inclusion of a scientific source were limited to the availability of information about the invented drugs and/or methods of their application, which allow due to physical and chemical factors of local interaction to urgently dissolve, discolor and remove from the surface of the skin, textiles or enamel stains of dried or thick blood and / or pus. The criterion of exclusion from the article was the lack of information about the invented means and / or methods providing urgent dissolution, removal of blood stains and pus from the contaminated surface and its bleaching with a local single application. The risk of individual bias in judgments was reduced by relying on the essence of the invention as a generally accepted criterion of novelty. A total of 29750 sources of information were examined, of which only 25 inventions were evaluated for consideration.

3. Results and discussion

The results of the analysis of the available scientific and patent information have shown that today there is no known means and no ingredient included in the composition of known combined preparations, the use of which separately from each other would ensure the rapid dissolution and loosening of biological stains bonded by coagulated proteins of animal, vegetable or microbial origin [11]. At the same time, the combination of hydrogen peroxide solution with alkali represents a surprisingly very promising direction for the development of new detergents, cleaning and bleaching agents. It is shown that in the last 20 years a whole Arsenal of new medicines and technologies of personal hygiene was invented, which allow for 1-2 minutes to completely clean the surface, stained with blood and/or pus [12,18]. The new drugs are designed for urgent dissolution, loosening, removal and discoloration of thick biological masses such as thick pus, sulfur plug, lacrimal stone and dry blood crust [11]. Such means are intended mainly for the treatment of purulent diseases (such as peritonitis, pancreonecrosis, pleurisy, otitis), thrombosis of vascular catheters and veins, as well as for discoloration of the skin and nail plates in the area of bruises and hematomas. The analysis showed that these drugs are aqueous solutions, and their main ingredients are hydrogen peroxide and sodium bicarbonate in certain ratios and concentrations [16].

Today it is safe to say that the development of a new group of detergents and bleaching hygiene products began in Russia [11]. The first means were developed for dissolving and removing thick pus from purulent wounds. In particular, the beginning of the patent for the first invention of this group of hygiene products begins officially with 29.12.2000. It was on this day that the patent application for the invention "Method of treatment of long-term non-healing wounds" (RU Patent 2187287) was registered. In this invention, for urgent cleansing of the wound from purulent necrotic masses, it was first proposed to irrigate the wound heated to 37 °C with a solution of 3% hydrogen peroxide, after which it was proposed to warm the wound surface to 42 °C. Then, in 2005, the invention "Method for treating pleural empyema cases" (RU Patent 2308894) was created. It was the second invention in which for urgent removal of pus from the pleural cavity it was recommended to introduce into the purulent mass a solution of an alkaline surfactant heated to 42 °C.

In 2006, patent applications were filed for inventions such as "Hyper-gassed and hyper-osmotic antiseptic mixture" (RU Patent 2331441) and "Method for peritoneal dialysis using gasified solution" (RU Patent 2336833). In these inventions as a solvent of thick pus, an aqueous solution of 2.7-3.3% hydrogen peroxide, 0.9-10.0% sodium chloride and gaseous carbon dioxide was proposed at an excess pressure of 0.2 ATM at +8 °C. The new drug had the ability to cold boil in the purulent mass, so it literally "blew up" a monolithic piece of thick pus.

In 2009 there was a publication about 3 inventions: "Softening agent for thick and viscous pus" (RU Patent 2360685), "Malchikov's method of removing bile calculus" (RU Patent 2367375) and "Methods of diagnostics and treatment of clotted hemothorax" by A. Y. Malchikov (RU Patent 2368333). They were proposed to dissolve thick pus, gallstones and blood clots with warm solutions of hydrogen peroxide and sodium bicarbonate.

These inventions created the basis for the modernization of developments in the future. And so it happened: in the following years, the first successes in the urgent washing of various surfaces from thick pus and blood stains due to physical and chemical dissolution and loosening of biological masses continued to improve. The list of new drugs has increased. Today we can assume that the results can be transferred to another field of activity. In particular, the experience of creating new cleaners for the field of medicine can be used to create new cleaners for enamel ceramic products used in the field of life. It is likely that the most useful may be the results achieved in our time in the development of a group of drugs, loosening blood crusts and dissolving and brightening blood stains.

The fact is that the most difficult to remove stains that occur on tooth enamel and ceramic dishes are formed due to coagulation, denaturation and/or drying of proteins and protein-lipid complexes [19]. Therefore, proteins play the role of a kind of biological glue that glues pigment particles into a conglomerate and firmly adheres it to the surface of the enamel. The sources of this natural "glue" are animal and vegetable products containing proteins and fats [4]. In addition to readymade proteins, fats and oils, similar products of microbial processing of starch, sugar, fiber and other substances also participate in the bonding of organic substances [19]. The fact is that food is not sterile. Is not sterile and the surface of the enamel. Moreover, very often foods are pre-seeded with fungi (yeast) for culinary purposes. It is also very important that the remains of food products after they are glued to the enamel always acquire a darker color, which rarely depends on the original color of the food. On the contrary, the transformation of color in an ordinary stain of enamel contamination is likely to be similar to the transformation of color in the mass of food eaten in the process of their movement in the gastrointestinal tract: the food eaten may consist of white products such as white bread, white starch, milk, cottage cheese and sugar, but the feces formed from them, normally acquire a dark brown color. In this regard, it is likely that the dark color of feces, plaque and stains on ceramic products (such as dishes) is determined by the microbiological process of processing food residues and the formation of conventional organic pigments.

It is no secret that the main pigment that stains feces and plaque in a dark brown color is the pigment stercobilin, which is formed as a result of enzymatic and microbial destruction of blood proteins and/or bile [17].

It is shown that the color of blood depends mainly on the color of red blood cells, the color of which, in turn, depends on the color of hemoglobin. It is believed that under the action of enzymes and microorganisms, hemoglobin is converted successively into verdoglobin, biliverdin, and then into bilirubin. Bile also contains the pigment bilirubin. Bilirubin is converted under the action of microorganisms first into urobilinogen, and then into stercobilin. Stercobilin is the pigment that gives "digested" foods a characteristic brown color [20].

It should be noted that people have long learned to remove from household items dark stains of animal and vegetable origin (they usually stain textiles, wool, paper, wood and leather). More than 100 years ago, wood ash and water passed through such ash were used for this purpose [21]. Today, specially designed washing powders and bleaching solutions are used for these purposes [22]. It was found that most of these solutions are aqueous solutions of hydrogen peroxide with special physical and chemical properties [23,24].

However, hydrogen peroxide has been used for more than 100 years for industrial washing and bleaching of various products of plant and animal origin, namely-wood, paper, textile, leather products [25]. At the same time, the safety of hydrogen peroxide for consumers of products bleached with this bleach was shown.

The experience of industrial application of hydrogen peroxide has shown that hydrogen peroxide is highly reactive, strong oxidizer and bleaching agent. It is shown that an increase in the concentration of hydrogen peroxide, an increase in the local temperature, an increase in the alkalinity of solutions above pH 7.0 and an increase in the duration of interaction enhances the bleaching effect of hydrogen peroxide. It was found that the bleaching effect of hydrogen peroxide increases the addition of compounds such as peroxosulfates, sodium peroxodisulfate, potassium peroxodisulfate, carbamine peroxide, ammonium peroxodisulfate, sodium carbonate, ammonia and silicate [23,24].

In addition, almost as many years hydrogen peroxide solutions are used for hair whitening.

In modern times, hydrogen peroxide is used to disinfect the water in which live fish swim [26], as well as to bleach seafood such as squid, octopus and cuttlefish [27]. It has been shown that fish remains safe for humans when ingested.

The fact that hydrogen peroxide is able to discolor various organic pigments, it became known to people also for a long time. In particular, in 1982, it was shown that the bleaching effect of hydrogen peroxide on hemoglobin is associated with hemolysis of erythrocytes and with the destruction of light-absorbing double bonds within colored pigments [28].

However, in the medical field, the first invention, which is a warm alkaline solution of hydrogen peroxide designed to discolor clothing stained with blood stains, was published in 2009. It was " a Method of rapid removal of blood stains from clothing" (RU Patent 2371532) [29].

Then in 2010, an article was published about a clinical case in which a urea gel containing 15% hydrogen peroxide minimized the discoloration and discomfort associated with ecchymosis [30]. It was shown that skin pigmentation in the area of ecchymosis was reduced due to the application of hydrated gel containing 5 to 20% hydrogen peroxide or urea peroxide to the skin during occlusion with an adhesive bandage.

In subsequent years, about 10 more drugs and methods were invented for urgent discoloration of the bloody bandage, skin and nail plate in the area of the wound, bruise and hematoma with solutions of 0.01-3% hydrogen peroxide and 1.7-10% sodium bicarbonate at a temperature of 37-42 °C [18]. Thus, during this period, the following inventions were developed: "Persons infrared differential express-diagnostics of bruising and injury of soft tissues" (RU Patent 2577510), "Bruise bleacher" (RU Patent 2539380), "Bleaching agent" (RU Patent 2589682), "Agent for intradermal bruise whitening" (RU Patent 2573382), "Method of skin discoloration in the area of bruising" (RU Patent 2582215), "Method for skin discoloration in bruising area" (RU Patent 2586278), "Method of removing paint from skin" (RU Patent 2600504), "Method for emergency bleaching and blood crust removal from skin in place of squeezed out acne" (RU Patent 2631593), "Means for intravital skin whitening near blue eyes" (RU Patent 2639485), "Method for whitening of bruise under eye" (RU Patent 2639283), "Bleaching opener of dried blood for wrapping bandages adhered to a wound" (RU Patent 2653465), "Decolorant of blood" (RU Patent 2647371), "Method for whitening of sore under nail" (RU Patent 2631592), "Method for blue nail treatment" (RU Patent 2641386) and "Method of emergency bleaching of the skin hematoma under the eye" (RU Patent 2679334).

The materials of these inventions prove that warm alkaline solutions of 0.1-3.0% hydrogen peroxide at pH 8.5 are unsurpassed leaders in bleaching fresh, dry and old blood.

Analysis of the composition of the invented bleach bruises and hematomas showed that the preparations of this new group of funds differ from all known detergents following properties [11]:

- 1. The main ingredients of bruise bleach are water, hydrogen peroxide and sodium bicarbonate.
- 2. Water, hydrogen peroxide and sodium bicarbonate are safe substances.

- 3. All bruise, hematoma and blood stain bleaches are alkaline oxidizers and have a pH of 8.5.
- 4. Bruising bleaches are applied locally at a temperature of 37-42 °C.
- 5. Water, hydrogen peroxide and sodium hydrocarbonate when absorption into the blood do not cause poisoning.

Independent studies show that solutions of hydrogen peroxide and sodium bicarbonate are fairly stable [31].

To date, it has been found that the mechanism of local action of alkaline bleaching oxidants is as follows:

- with local action, they have the ability of alkaline saponification of proteins and protein-lipid complexes,
- they have the ability to destroy biological masses from within due to the process of cold boiling
- they have the ability to destroy the light-absorbing double bonds inside the colored pigments.

It is known that hydrogen peroxide solutions as a bleaching drug are safe for people [32]. Therefore, a solution of 3% hydrogen peroxide is sold in pharmacies without a prescription as a local antiseptic, a solution of 5% hydrogen peroxide is used for hair whitening, a solution of 6.5% urea peroxide is used as an over-the-counter softener of earwax, and a solution of urea peroxide at a concentration of 5-20% is widely used in dentistry for teeth whitening [33,34]. In turn, sodium bicarbonate is an edible substance and is used in cooking under the name "baking soda".

It should be noted that following the discovery of thick pus solvents and bleach bruises and blood stains were invented tools that can completely remove plaque from the surface of the teeth and ceramic dental structures in a few tens of seconds after the start of local interaction. So, in 2017, a patent for the invention "Frictional toothpaste" (RU Patent 2626669) was issued. In this invention, for the urgent removal of dental plaque, a solution of 9.5-10% sodium bicarbonate and 0.5-1.5% hydrogen peroxide was first proposed, which in a ratio of 5/1 mass is poured into crystalline sodium bicarbonate at a temperature of +25 - +26 °C. As a result, a thick paste is formed, in which soft sodium bicarbonate crystals have abrasive, frictional, adhesive, erasing, absorption and adhesive activity against the rough and soft surface of the "tooth" dirt, but not against the smooth and hard surface of the tooth enamel and the smooth surface of the gum mucosa. On the other hand, the solution due to the dissolved part of sodium bicarbonate causes alkaline hydrolysis of proteins and saponification of fats, and due to hydrogen peroxide as a result of the catalase reaction releases oxygen gas, the bubbles of which cause the effect of cold boiling and destruction of biological masses.

Then in 2018, a patent was obtained for the first "Bleaching cleanser of dentures" (RU Patent 2659952). The invented bleaching agent is an aqueous solution of 3% hydrogen peroxide and 2-10 % sodium bicarbonate, which contains oxygen gas under an excess pressure of 0.2 ATM at +8 °C. Before use, this drug is heated to a temperature of 37-42 °C. It is shown that the developed drug in a few seconds after the beginning of local interaction with dirty teeth, dentures, ceramic utensils and surgical instruments completely and automatically

cleans, deodorizes and whitens their surface. Purification and bleaching is achieved by hyper temperature softening, alkaline saponification, cavitation loosening, dissolution and oxidative discoloration of various biological masses.

Thus, heated to 42 °C and saturated with oxygen gas under excessive pressure, aqueous solutions of 0.01-3% hydrogen peroxide and 1.7-10% sodium bicarbonate are automatically converted into strong detergents and bleaching agents. Such means are capable at single application within one minute automatically to clear enamel from a dental plaque, the rests of food, traces of pus, blood stains and from pollution by other biological products. At the same time, these bleaching agents remain safe. What's more, they are edible! (*Fig. 1*)



Fig. 1 Oxygen, used as a bleaching agent 1. ábra Oxigén, mint fehérítőszer

4. Conclusions

From the end of 2000 to the present time, more than 20 drugs and methods of their application in the medical field have been invented for emergency dissolution and discoloration of dense biological masses bonded by protein and protein-lipid complexes of blood and pus as a result of their coagulation and/or drying. Analysis of the composition of these preparations showed that all of them are aqueous solutions of 0.01-3% hydrogen peroxide and 1.7-10% sodium bicarbonate, heated to a temperature of 37-42 °C. It is shown, that the additional introduction of oxygen gas, carbon dioxide or other gases under excessive pressure, or insoluble sodium bicarbonate powder enhances and accelerates the washing and bleaching effect of these mixtures. It is established, that the mechanism of washing and bleaching action of such mixtures is that they have alkaline saponification of protein and protein-lipid complexes, the formation of gas bubbles at cold boiling and oxidative destruction of lightabsorbing double bonds inside the colored pigments. It is proposed to use the experience of development and the existing Arsenal of new medical alkaline bleaching cleaners blood stains and pus for the analysis and development of new effective and safe bleaching cleaners ceramics in the home.

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