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RECOVERY OF WASTE LEATHER FROM TANNERIES AS AGGREGATE IN COMPOSITE MATERIALS

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ABSTRACT. This study proposes a recovery method for waste leather from tanneries, with high chromium content, by incorporating it into a composite material with cement matrix. The natural aggregates were substituted with CRT (cathode ray tubes) glass waste, with high lead content, originated from televisions and monitors dismantling. The material technological production flow was as follows: grinding skin (two types of skin: black and brown were used), mixing the ingredients after own recipes, pouring the mixture into molds, maturation, demoulding.

The new material obtained was submitted to leachability tests to determine, using AAS (Atomic Absorption Spectrometry), the total chromium and lead concentration released from the material. The results showed that Cr and Pb were well retained in the cement matrix. The low levels of total Cr and Pb concentration were recorded at pH = 9-10 and pH = 12, for the composite containing black leather waste and and at pH = 2-3 and pH = 12 for the composite containing brown leather waste. Mechanical tests were also provided to evidence the mechanical properties of the composite. The new composite material obtained may be considered as construction material and can be classified in mortars class because of the size of the used aggregate (CRT glass waste).

The objective of this study was to obtain a composite material that can be used as material for construction, in compliance with current standards in construction and environmental legislation.

The proposed method for leather waste recovery is viable and environmentally friendly and could bring economic benefits.

Key words*: leather, waste, CRT, composite, material.*

INTRODUCTION

The leather industry production and processing sector generates high quantities of tanned leather waste, whereby, an important amount reaches the municipal landfills.

The leather is an intermediate industrial product with numerous applications in various sectors. Globally are processed annually approximately 5.5 million tonnes of wet salted hides, of which are produced about 460,000 tons raw leather and 940 million square meters of finished leather.

It is worth to mentioning that in EU, in 2006 leather processing sector reached about 3,700 enterprises (Eurostat, 2009). These are small and medium-sized family tanneries. European tanners are dependent on raw material from outside the European continent. In recent years, this sector has a downward trend since the Asian and American market experienced a significant development.

Europe annually produces about 74,000 tons of raw leather and approximately 240 million square feet of finished leather. Leather processing generates large amounts of waste. Thus, after processing 1,000 kg of wet salted skin were obtained only about 200 kg of usable leather, remaining 800 kg of waste, such as: waste tanned leather (about 250 kg), waste leather untanned (350 kg) and wastewater (about 200 kg). The amount of required water is about 45-50 m^3 to 1,000 kg of processed leather. Also, the consumption of chemical reagents used for the tanning of 1,000 kg of wet salted skin is very high, an amount of about 400 kg of chemicals, such as: sodium chloride, lime, sodium sulfide, sulfuric acid, chromium sulfate, etc (BAT, 2013).

Because the factories produce various leather items, they produce significant amounts of wastewater and used chemicals (Famielec et al., 2011), and these mostly arrive at municipal landfills and are considered as hazardous waste (Regulation 301, 2014; Roşu et al., 2015). The quantity of such waste should be reduced because biodegradation is very slow and the emissions of chemicals into the environment are important.

Recovery of waste leather can be made by replacing the toxic chemical auxiliaries; find alternatives to chrome tanning; by recovery and recycling of solid waste (Kurian et al., 2009), but also is a goal of “clean technologies” because only 25% of raw skin is found in finished products (Ollé et al., 2013). Worldwide, about 70% of proteic waste are recovered, the rest being undertaken research to find solutions for use (Yilmaz et al., 2007). Waste from buffalo leather, 70% chromed is used mixed with soft leather and hard leather waste shredded to obtain artificial sole (Petrescu, 2011).

For fully and effective recovery of the solid waste from processing and manufacturing of leather, actions must be directed to: find new areas of recovery by processing; by physical-mechanical processing to obtain building materials or synthetic polymers mixtures for various uses; by physico-chemical processing in order to obtain chemical auxiliaries; incineration with heat recovery, which is a more efficient method in terms of environmental protection than landfilling them on the ground (Jing et al., 2011).

In leather industry, European Union practices the following waste recovery methods:

▪ Obtaining biofuel from waste from fleshing skins (pre-tanned). These wastes are mostly animal fats and meat pieces remaining on the skin. This process provides a washing stage, to remove dirt and blood, then a heating one to a temperature of 110°C, afterwards a filtration to remove the remaining pieces of meat. After these steps, the waste fat obtained is mixed with an acid and a catalyst to give the methyl ester (Alptekin et al., 2012; Onga et al., 2013).

▪ Biofuel production by pyrolysis in a fluidized bed reactor at a temperature between 450- 600°C, use the leather waste in post-tanning stage (finishing leather shavings, chips) (Yilmaz et al., 2007).

The purpose of this study was to obtain a composite material containing a mixture of leather waste with chromium content and CRT (catode ray tubes) glass waste (cone) with a high content of lead, as agregate, to replace the natural agregate and to provide an alternative for the disposal of this waste, which can be used as a construction material.

Also, one of the objectives was to study the metal (Cr and Pb) concentration from the leachate resulted by immersing the composite material in various solutions, simulating the behavior of this material in different environmental conditions.

About CRT (catode ray tubes) glass waste, various studies (Popovici et al, 2013[a]; Popovici et al, 2013[b]; Corbu et al., 2014; Corbu et al., 2015; Popovici et al., 2015[a]; Popovici et al., 2015[b]; Pop et al., 2016, Popița et al., 2016) have shown that this waste can be successfully used to replace natural aggregate in mortars used in construction.

MATERIAL AND METHOD

To achieve the study objective was used a recipe for the composit material based on clasical mortar recipe (cement matrix, water, aggregates) replacing natural aggregates (sand) with waste leather (two types: black and brown) and CRT glass waste.

For the preparation of the composite material was used white cement CEM_II_A-L_52.5N (used to obtain high-performance concretes) Portland Cement Composition (80 to 94%), additions - limestone (6 ÷ 20%), minor component (0 to 5%); initial setting time (≤ 5 min) (Lafarge, 2013).

In the beginning the leather was grinded. The grinding device IKA A11 basic can be used for different procedures: grinding of hard, brittle soft fibrous or non-elastic materials; with Mohs hardness of up to 6 (IKA).

The composite material was prepared by mixing: cement matrix, water, waste leather (14%) and CRT glass waste (14%). For this study were used to types of leather waste: black and brown.

For the chemical analysis were needed small samples and the homogenized material was poured into a mold 2 cm^3, allowed to cure three days and then peeled.

Chemical analysis

In a first stage the samples were immersed in different pH solution and ultrasonicated to activate the metal leaching; in the second stage the leachate was analyzed to detect chromium and lead that could migrate of the composite material structure.

The different pH solution was made with distilated water by adding HNO_3 and NaOH. The obtained solutions had pH=2-3, pH=5-6, pH=9-10 and pH=12. The ratio liquid/solid was chosen according to OM 95 (2005).

Afterwards, the samples were immersed and were ultrasonicated for 30 minutes at room temperature (20°C).

In figure 1 is presented the preparation for the ultrasonication and the ultrasonic bath (S 10 H Elmasonic) used.

Fig. 1. *Preparation for the ultrasonication and the ultrasonic bath S 10 H Elmasonic*

The ultrasonic bath (S 10 H Elmasonic) has dimensions of 190x85x60 mm and the capacity is 0.8 L. Operating frequency is 37 kHz and uses a propagation system of waves type sandwich. This model has more features: the heating function, the self-degassing and sonication autostart when the desired temperature is reached (Elmasonic).

After ultrasonication, the leachate was filtered and the pH was adjusted to a pH=2 to can analyze the leachate samples by absorption atomic spectrometry (ZEEnit 700 by Analytik Jena), used for metal analyses (flame method for concentration of mg/L, error ± 5%).

The obtain results were demonstrated that metals (Cr and Pb) concentrations in leachate was within acceptable limits or even lower.

Physical and mechanical tests

Designing and manufacturing the mortar mixtures were conducted to demonstrate the ability of the construction material, namely mortars, to integrate / embed successful hazardous waste type (leather with Cr content and CRT glass with Pb content) retaining its mechanical properties up to standard limits.

The used materials for manufacturing the samples for the mechanical tests were: cement, leather waste type: black, CRT waste glass (cone) and water of the drinking water source of the city.

It was performed the first blank recipe (M) / benchmark for mortar with gray cement CEM I 42.5 R Potland, according to SR EN 196-1 standard profile: 2006 Methods of testing cement. Part 1: Determination of mechanical resistance.

In the second blank recipe, (M_A) has replaced the Potland cement CEM I 42.5 R (gray) with a white cement type CEM II A-L 52.5N to highlight used waste in mixtures to their installation and subsequent finishing.

The third recipe (M_{AST}) was carried out by the complete replacement of river sand used in conventional / ordinary mixtures with glass with maximum diameter of 4 mm.

In the fourth recipe (M_{ASTPG}) the natural aggregate (sand) was replaced with CRT waste glass (94%) and grinding leather waste type black (6%).

It is worth to mention that compared with ultrasonic procedure where a higher percentage of waste leather was embedded to observe the metal migration from the composite material structure, for the mechanical tests was respected the aggregate proportion established by standards for building materials.

Technical characteristics of gray cement CEM I 42.5R are: Portland clinker (95 ÷ 100%) minor component (0 ÷ 5%) initial setting time (60 min), flexure strength at 2 days (minimum 20 N/mm^2); flexure strength at 28 days (42.5 < x ≥ 62.5 N/mm^2) (Holcim, 2013).

In figure 2 are presented the four recipes prepared for foundry casting.

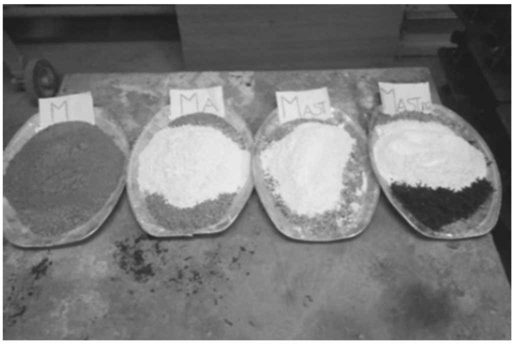

Fig. 2. *The four recipes prepared for foundry casting*

After completing the milling stages, weighing, and placing the material in tray, was passed to molding the composite. It was molded in standard moulds with the cross-section of 40 mm × 40 mm and a length of 160 mm, with stable walls by approximately 10 mm thick, were previously smeared with oil avoid joining fresh mass of the composite.

After mixing and pouring every composite and casting in moulds a vibrating table was used to push up to the surface of the material the air bubbles and water. The mixtures of the four composite molded into standardized moulds (40x40x160 mm) are presented in the figure 3.

Fig. 3. *Mixtures of the four composite molded into standardized moulds (40x40x160 mm)*

The samples were peeled after curing 24 hours, then weighed, labeled and immersed in water at room temperature until the age test (2, 7, 28 days). At dipping into the water basin the samples were placed on a rubber mat nicked so samples are separated and water can get free on the six faces.

The mechanical tests used for this study were: flexural strength test and compressive strength test. The equipments used for the mechanical tests, are hereunder described:

Flexural strenght test was made with a Technotest device. Before the flexural strength test the samples were weighed. The flexural load was applied vertically, with a 50 N/s until the material failed. Normally rupture should occur within a period of 30-90 seconds. Attempts were compliant to SR EN 196-1: 2006.

Compressive strength test was made also with a Technotest device. In the test device was placed in turn half an angle prism obtained from the flexural strength test. Following compression applied prism halves were broken into 2-3 pieces.

RESULTS AND DISCUSSION

Interpretation of the chemical analysis results

In Table 1 is shows the obtained results for total chromium and lead concentrations from leachate analysis (black waste leather) using AAS ZEEnit 700 Spectrometer by Analytik Jena, flame method (without atomic speciation).

Table 1. *The influence of pH on total Pb and Cr concentration for the composite with black and brown leather waste content*

Composite with leather	**pH**	**Cr_{total} (mg/kg)** determinated	**Cr^{*}_{total} (mg/kg)** legislation	**Pb_{total} (mg/kg)** determinated	**Pb^{*}_{total} (mg/kg)** legislation
Black	2 - 3	**0.53**	0.20	**2.12**	0.20
	5 – 6	**0.31**		**1.89**	
	9 - 10	**0.31**		*BDL*	
	12	**0.42**		0.06	
Brown	2 - 3	**0.39**	0.20	*BDL*	0.20
	5 - 6	**0.92**		**4.83**	
	9 - 10	**0.71**		**7.48**	
	12	**2.35**		*BDL*	

**MO 95/2005 – MAC – Maximum Allowed Concentration*
**BDL - Below Detection Limit*

From Table 1 the leachate of the black leather composite at a pH=2-3 and pH=5-6, the total Cr and Pb concentrations exceeded the MAC (maximum allowable concentration). At a pH=9-10, the total Cr concentration has an identical value to that obtained for a pH=5-6, so very close to MAC, however, the total Pb has concentration values below the detection limit. At pH=12, the total Cr concentration of 0.42 (mg/kg) exceeds two times the MAC, and the total Pb concentration value of 0.06 (mg/kg) is well below MAC.

The obtained results for the leachate of the composite material with brown leather waste content at pH=2-3, the total Cr concentration exceeds two times the MAC and total Pb is BDL. At pH=5-6 and pH=9-10 the total Cr and Pb concentrations values were significantly above MAC. At pH=12, the total Cr concentration exceeds 12 times the MAC, but total Pb concentration is BDL.

Interpretation of the physical-mechanical tests results

For all determination were made three samples (the moulds are designed for three samples), were weighted and the density and average density were calculated (table 2). Mechanical determinations: determination of flexural strength and determination of compressive strength.

Table 2. *Weight and density of samples after stripping*

Sample name 40x40x160mm	Sample number	Weight (g)	Density D=W/V ($kg•m^{-3}$)	Average density ($kg•m^{-3}$)
M-blank	1	577.9	2,257.42	**2,261.46**
	2	581	2,269.53	
	3	577.9	2,257.42	
M_{A}-blank with white cement	1	568.1	2,219.14	**2,223.18**
	2	572.5	2,236.33	
	3	566.8	2,214.06	
M_{AST}-CRT glass waste	1	628.4	2,454.69	**2,439.97**
	2	624.5	2,439.45	
	3	621	2,425.78	
M_{ASTPG}-Leather (type black) and CRT glass waste	1	560.9	2,191.02	**2,226.17**
	2	574.6	2,244.53	
	3	574.2	2,242.97	

Once samples have been peeled, each one was weighed separately. It was observed that higher weight had the M_{AST-}glass samples and the lowest the samples M_{ASTPG} + glass-leather.

After the test age (2, 7 and 28 days) the samples were weighted again (table 3), and it can be observed that again the higher weight had the M_{AS-}glass samples and the lowest the samples M_{ASP} + glass-leather.

The density (average) is higher than 1,300 kg•m^{-3} which mean that the samples overdraw the light mortars.

Table 3. *Weight and density of samples after age tests*

Sample name 40x40x160mm	Sample number	Age test (days)	Weight (g)
M-blank	1	2	**581.2**
	2	7	**598.2**
	3	28	**600**
M_A-blank with white cement	1	2	**575.2**
	2	7	**590.1**
	3	28	**598**
M_{AST}-CRT glass waste	1	2	**632.4**
	2	7	**640.5**
	3	28	**650**
M_{ASTPG}-Leather (type black) and CRT glass waste	1	2	**568.7**
	2	7	**592.7**
	3	28	**600**

Flexural strength interpretation results

Flexural strength tests were performed according to SR EN 196-1:2006, as follows: the prism is placed in the flexural strength device with the lateral side of the sample on the roll support and with the longitudinal axis perpendicular to the support. Vertical load applied by load roll on the opposite side of the prism and constantly increase the load with (50 + 10) [N/s] to failure. Half prism is maintained until their compressive strenght test.

Flexural strength, Rf, was calculated by the following formula (according to SR EN 1015-11 /2002):

$$\mathrm{Rf} = \frac{1.5\times \mathrm{Ff}\times \mathrm{l}}{\mathrm{d}\times \mathrm{b}^2} \qquad [1]$$

where: Rf = flexural strength [MPa] or [N•mm^{-2}]; d = square section prism side [mm]; b = length of the sample [mm]; Ff = applied load in the middle prism at breaking [N]; l = distance between supporting rolls [mm].

Taking the samples M and M_A as blank and comparing them with standard limits, it can be said that samples containing CRT glass waste (M_{AST}) and glass + leather waste (M_{ASTPG}) have values close to the blank values. The sample M_{ASTPG} has the flexural strength values under the blank values, but closer.

According to SR EN 1996-3 Eurocode 6, the characteristic flexural strength values range between 0.2-2 [MPa, N·mm^{-2}].

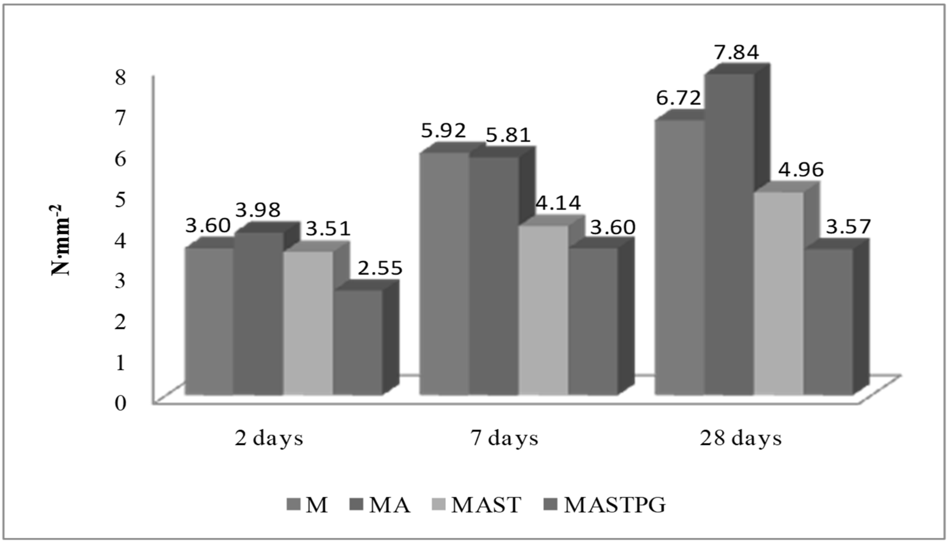

Fig. 4. *Flexural strength of the composite samples*

From figure 4 can observe that the obtained composite material had good mechanical properties above the flexural strength standard values.

Compressive strength interpretation results

Compressive strength tests were conducted according to SR EN 196-1: 2006. It was attempting prism halves from flexural strength to the lateral sides of the sample. It crosses every half prism side relative to the platters/ plates of the machine with accuracy ±0.5 mm.

The compressive strength Rc was calculated with the formula 2 (according to SR EN 1015-11 /2002) as follows:

$$Rc = \frac{Fc}{1600} \quad [2]$$

where: Rc = compressive strength [N•mm^{-2}]; Fc = maximum load when prism is breaking [N]; 1600 = platters area [mm^{2}].

As was observed and demonstrated by various researchers (Ling et al., 2011; Moncea et al., 2012), the compressive strength decreases with increasing the amount of waste CRT glass, the results were confirmed for samples (M_{AST}) containing CRT glass waste and for the samples (M_{ASTPG}) with glass + leather waste the compressive strength decreases two times comparative with M_{AST} results (figure 5).

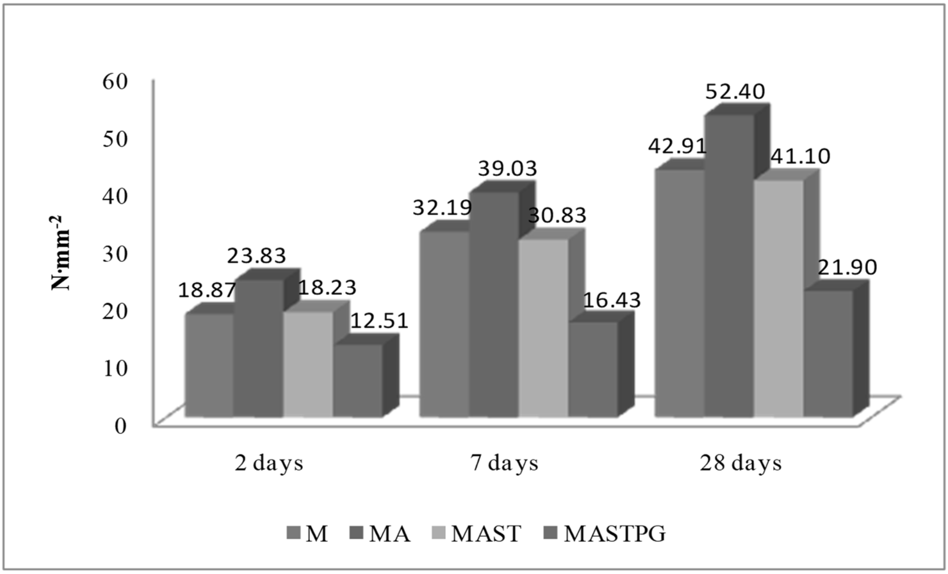

Fig. 5. *Compressive strength of the composite samples*

The obtained results for compressive strength were comparated with the values from SR EN 998-1: 2011 for plastering mortars and shown in Table 4.

Table 4. *Comparison of the obtained results compressive strength with standard values for plastering mortars*

	Compressive strength (28 days)	
Mortar class	**Standard values [N·mm^{-2}]**	**Experimental obtained values [N·mm^{-2}]**
CS I	0.4-2.5	21.9 - 52.04
CS II	1.5-5	
CS III	3.5-7.5	
CS IV	≥ 6	

All obtained values exceed the compressive strength for plaster mortars, and the composite material may be classified in mortar class CS IV.

Also, the obtained results for compressive strength were comparated with the values from SR EN 998-2:2011 for masonry mortars and presented in table 5.

Table 5. *Comparison of the obtained results compressive strength with standard values for masonry mortars*

	Compressive strength (28 days)	
Mortar class	**Standard values [N·mm^{-2}]**	**Experimental obtained values [N·mm^{-2}]**
M1	1	21.9 - 52.04
M2,5	2.5	
M5	5	
M10	10	
M15	15	
M20	20	
Md	d; d≥25	

The experimental obtained values offered the possibility of framing the composite obtained in class M20 and Md. The mortar containing waste glass and leather can fit in M20 class.

From the above tables we had obtained a material with good mechanical properties which can be proposed for use in building construction and can be used to produce of decorative elements, joints.

CONCLUSIONS

To propose practical applications for a composite material containing waste (CRT glass and leather) by replacing natural aggregate (sand) was studied the leachability mechanisms of total chromium and lead from material immersed solutions with different pH. From the obtained results can conclude that:

- low levels of total Cr concentration were recorded at pH = 9-10 and pH = 12 for the composite containing black leather waste and at pH = 2-3 for the composite containing brown leather waste.
- low levels of total Pb concentration were recorded at pH = 9-10 and pH = 12 for the composite containing black leather waste and at pH = 2-3 and pH = 12 for the composite containing brown leather waste.

The obtained values for the other pH concentrations exceed the MAC from legislation, but the values are not very high, further studies are needed to improve the recipe with other material to chelating the two metals.

After studying the mechanical behavior of the composite material can be made the following statements:

- new composite material obtained may be considered as construction material and can be classified in mortars class because of the size of the used aggregate (glass).
- the embedding of the leather waste together with CRT glass waste, was successful with good results for the flexural and compressive strength.

The composite may be used to produce of lightweight building elements, decorative objects, such as: embroidered decorative elements for facades, decorative elements for interior, decorative elements for parks and gardens.

Manufacture of a composite containing CRT glass and leather waste, can reduce the waste CRT glass and leather quantities, leading to solve an important environmental protection objective, namely reducing/eliminating landfilling of these types of waste.

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