Scientific report

2017

Development of ecofriendly composite materials based on geopolymer matrix and reinforced with waste fibres

Contents

1. Project overview

The project is an answer for a specific challenge connected with waste management, recycling and urban mining. The main objective of the project is to prepare a broad spectrum of advanced and progressive new fibre-based materials for construction industry with high potential of commercial utilization, especially development of composite materials from waste natural fibres such as animals and vegetables, for replacing the traditional construction materials.

The consortium of the project is composed of universities from countries situated in Europe and South America. The project members are: Cracow University of Technology, Poland (project coordinator), Riga Technical University, Latvia, Omer Halisdemir University, Turkey, Babeș-Bolyai University, Romania, Politehnica University Timisoara, Romania, Pontificia Universidad Católica del Peru, University of Mar del Plata, Argentina and Catholic University of Uruguay Damas Antonio Larrañaga.

2. Objectives

The project has five objectives, denoted as "Work Packages":

Work Package 1. The selection of waste materials (fly ash) to be hydrothermally alkalized and therefore turned into new material based on geopolymer matrix for construction application

Aim: Preparation of the theoretical concept for the new composites, analysis and optimization of the structure and mechanical properties and performance of designed composite materials and assessment of their materials for selected applications

Participants: Omer Halisdemir University (coordinator), Babeș-Bolyai University, Cracow University of Technology, Pontificia Universidad Católica del Peru, University of Mar del Plata

Deadline: 29 December 2017

Work Package 2. The selection of waste materials (natural fibres) as a fillers and therefore turned into new composites for construction application

Aim: The selection of most suitable waste fibres added to the hydrothermally alkalized fly ash in order to improve its properties

Participants: Babeș Bolyai University (coordinator), Cracow University of Technology, Pontificia Universidad Católica del Peru, Riga Technical University, Catholic University of Uruguay Damas Antonio Larrañaga

Deadline: 31 March 2018

Work Package 3. Optimization using computer methods properties of new materials and structural elements made of them

Aim: Analysis and optimization using computer methods of the structure and mechanical properties of composites and the assessment of their ability to be used the construction materials.

Participants: Riga Technical University (coordinator), **Politehnica University Timisoara**, University of Mar del Plata

Deadline: 30 June 2018

Work Package 4. The research into the application of new materials – comparison functional properties materials

Aim: Comparison between the new composites and the traditional materials in regard of their properties in laboratory

Participants: Pontificia Universidad Católica del Peru (coordinator), Cracow University of Technology, **Politehnica University Timisoara**, Riga Technical University, Catholic University of Uruguay Damas Antonio Larrañaga, University of Mar del Plata

Deadline: 30 June 2019

Work Package 5. Analysis of practical applications of new materials for construction application and testing prototype components in laboratory as well as validated it in relevant environment

Aim: Preparation of solutions and testing prototype components in lab as well as in relevant environment and comparison between the new composites and the traditional materials in regard of their properties in varying environmental conditions.

Participants: Cracow University of Technology (coordinator), Babeș-Bolyai University, **Politehnica University Timisoara**, Omer Halisdemir University, Riga Technical University, Catholic University of Uruguay Damas Antonio Larrañaga

Deadline: 27 December 2019

3. Results

For 2017, the project objective was, for each participant involved in the WP, to research the potential waste products that can become candidates for the matrix of the new composite materials that will be designed and manufactured as part of other work packages.

3.1. An overview of the available geopolymer sources in Peru

The construction industry in one of the biggest and most influential industries worldwide, its contribution towards the global GDP revolves around one-tenth of the total amount [1]. From the environmental point of view, the construction industry accounts for more than 30 % of total carbon dioxide emissions. Comparing to other economic activities, the global construction industry consumes more raw materials (about 3000 Mt/year, almost 50% by weight) [2]. Therefore, the replacement of conventional construction materials, i.e. ordinary Portland cement (OPC), with the use of geopolymers to develop a new cementitious material is being broadly investigated [3-4]. Peru is not an exception of this reality. The construction industry in the last decade has grown very fast with an average rate of 10% per year (a maximum rate of 17% was achieved in 2010). The use of construction materials from recycled sources in this context is of high interest.

Available sources for potential geopolymer production in the country come from: the recycle process of fired clay bricks used in buildings that are being demolished, and natural pozzolana from volcanic ash, readily available in the mountains in the southern part of the country.

In Peru, fired clay bricks are one of the most common materials for modern construction, which involves also hundreds of houses self-constructed in the outskirts of urban areas using bricks as its main material. With the developing and renovation of urban areas, a large amount of waste clay bricks (WCB) has been produced [6]. WCBs are typical silicate solid waste mainly consisting of SiO_2 and Al_2O_3 and the amount of the two components account for no less than 80 % of the total. Other compounds are Fe_2O_3, TiO_2, CaO, MgO among other metal oxides [5]. Due to their lower apparent density than other natural aggregates, and sharing some characteristics of other lightweight aggregates, WCB aggregates have also been used to produce closure walls in new reinforced concrete buildings. Still, recycling and application of WCBs is nonexistent in our country. Possible reasons for this may be due to their high porosity, high water absorption rate and great variation in quality, nospecifications of the material and uncertainties on regular supply and lack of confidence and experience in its use [6]. On the other hand, natural pozzolana (NP) are alumino-silicate materials from volcanic rocks. Extraction of NP in several deposits of Peru has grown exponentially in recent years [7], mainly as a replacement of Portland cement (up to 40% by mass) [7-8], however, it has never been used to form geopolymers by itself.

3.2. An overview of the available geopolymer sources in Turkey

Cement production involved the mining of raw materials such as sand, limestone and clay needs large amounts of raw materials and energy. Because of huge demand for concrete using ordinary Portland cement, the cement industry becomes a reason of environmental concerns. This situation is particularly due to high emissions of CO2 consisted of the burned of fossil fuels, alongside the decarbonation of limestone in the clinker production and depletion of natural resources [9]. CO2 emission into the atmosphere from the production of cement is approximated as 6% of the total greenhouse gas emissions [10]. Therefore, using waste materials for the construction industries has a promising future due to the increasing interest and the need for recycling waste product. Geopolymer (GP), an environment friendly inorganic binder is a developed alternative binder for concrete that utilizes industrial by-products. The geopolymer binder is produced by the reaction between the base materials which are affluent with silicon and aluminum with the alkaline solution [11]. So, the waste materials derived from industrial products such as fly ash (FA) is used for GP production to minimize the carbon footprint of cement productions. The reaction between the FA and the alkaline solution produces the geopolymer concrete in which the concrete ingredients consist of bonds. Although almost 15 million tons/year of FAs are generated from thermal coal-fired power plants in Turkey, a very small amount of it is utilized in the construction industry [12].

It was determined in Zeybek’s (2009) study that compressive strengths of geopolymeric bricks produced using class C FA of thermal plant were between 8 and 13 N/mm2 (MPa). Only 7 day compressive strengths were measured 11, 12, and 11 MPa, respectively, in geopolymer bricks produced as a result of heat treatment in the oven of 60 °C at 2, 24, and 72 h to Class C FA-sodium hydroxide-sodium silicate mixture [13].

As a result of measurements of the study of Doğan-Sağlamtimur et al. (2016), the best resulting geopolymer concrete products were obtained at a baking temperature of 150 °C with maximum 11.26 MPa compressive strength and at a baking temperature of 100 °C with maximum 10.82 MPa compressive strength from the class F FA samples activated with sodium hydroxide and sodium silicate activator, respectively [14].

The raw materials, which are used for this project, are supplied from two large-scale international firms from Turkey:

- Type 1 FA: Çatalağzı Termal Power Plant
- Type 2 FA: Isken Sugözü Termal Power Plant

Physical and chemical characteristics have important implications on the potential for productive reuse and reaction potential of these waste materials. In the study, SEM, XRD, XRF, DTA/TG and BET analysis of FAs will be determined. Then, to obtain some mechanical and physical properties of GP, compressive strength, flexural strength, freeze-thaw resistance, water absorption, porosity and moisture content will be tested on geopolymer material.

3.3. An overview of the available waste fibre sources in Uruguay

Preliminary studies in Uruguay dictated the given the quality and amount of raw cellulose waste at disposal, and the addition of new waste material sources. The Materials Engineering Group from Catholic University of Uruguay, School of Engineering and technologies is proposing matrix precursor materials that will be combined with waste and new reinforcements. The main waste materials sources to be analyzed are paper mill sludge (Primary, Bio and Precipitation) from paper mills and rice hull ash (RHA) from power generation plants.

Between 2004 and 2013 Direct External Investments in wood harvesting, wood production and paper mill factories summed over U$S 4.000 million (70% of that are paper mills). Currently, in Uruguay the paper mills industry generates 2.600.000 Tons of blanched cellulose pulp per year, with exports associated amounting over U$S 1300 million in 2015. The Elemental Chlorine Free process in those plants results in more than 35.000 tons a year of wet Primary Sludge and a similar or bigger amount of Bio and Precipitation Sludge. The amount and quality of those waste materials will determine if they're suitable for reinforcement or matrix manufacture, as will be discussed later.

Uruguay rice production amounts for total 1.125.000 Tons per year (full grain), obtained from 200.000 hectare dedicated land (up to 8 ton per hect.), of which 95% is exported. The industrial rice processing results in the production of 220.000 tons per year of rice hull, which is almost entirely used for power generation in biomass plants. Almost 25% of incinerated hull is inert and as result, are generated more than 55.00tons of RHA per year, that are mainly composed of amorphous silica (80-90%). A small proportion of this waste is used as aggregate of OPC and the rest is disposal in landfill.

3.4. An overview of the available geopolymer sources in Romania

Fly ash, also known as flue-ash, is one of the residues generated in combustion, and comprises the fine particles that rise with the flue gases. Ash, which does not rise, is termed bottom ash. In an industrial context, fly ash usually refers to ash produced during combustion of coal. Fly ash is generally captured by electrostatic precipitators or other particle filtration equipment before the flue gases reach the chimneys of coal-fired power plants and together with bottom ash removed from the bottom of the furnace is in this case jointly known as coal ash. Depending upon the source and makeup of the coal being burned, the components of fly ash vary considerably, but all fly ash includes substantial amounts of silicon dioxide and calcium oxide, both being endemic ingredients in many coal-bearing rock strata. Romania had 31 thermo-power plants (Fig. 1) [15]. In Gorj county from Romania is produced electricity in two thermal power plants: Turceni, with 2310 MW installed capacity and Rovinari with 1320 MW installed capacity. Both thermo power plants use the lignite as fuel extracted from mining basin of Oltenia. During technological process of coal combustion in boilers, ash and slag results separately. Ash and slag deposit is formed by solid part decanting (ash and slag) under gravitational effect, behind of basic embankments and cant embankments. The ash and slag are exhausted by hydraulic system in two deposits of slag and ash no.1, at 3.3 km distance from thermoelectric power plant. That means 52.64 ha, the volume of deposited ash being of 13.034.470 m^3. The area of land affected by compartment no. 2 of the same deposit is about 45.4 ha, and the volume is 10.825.795 m^3 [16, 17].

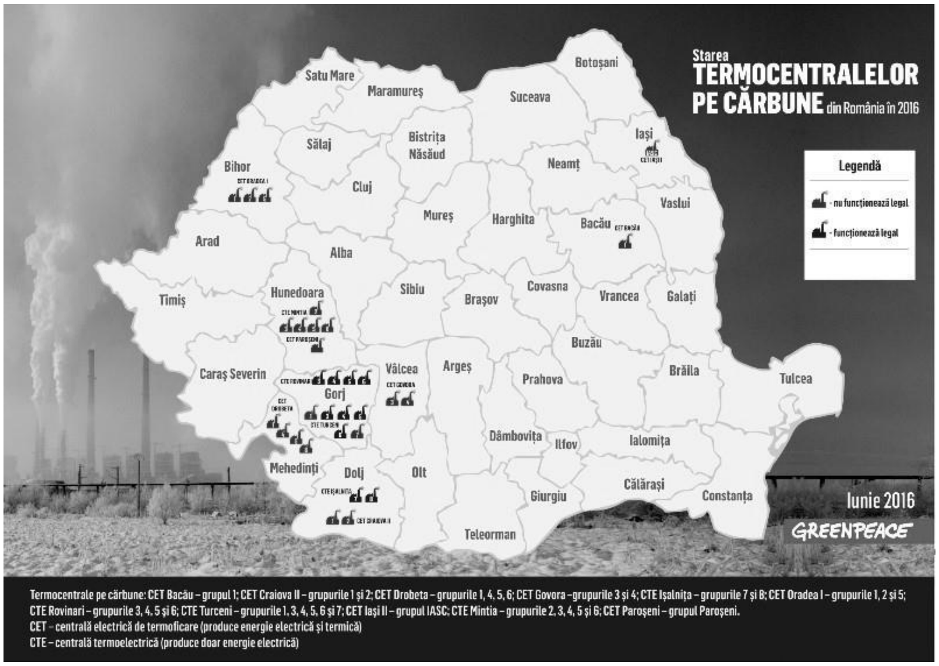

Figure 1. Thermo-power plants from Romania

The chemical composition of ash from main eight Romanian thermo-power plants (Table 1) showed that in all types of ash the main components are: SiO2, Al2O3, Fe2O3, their total exceeding 70%, which showed the possibility of using ash as a filler substitute for clay in the raw mix [18, 19].

Fly ash resulted from coal burning is a waste that can be used in geopolymer binder production. Romania has big deposit of fly ash, collected from the central heat and power plant. These big deposits of fly ash in Romania are not used and increase every year.

Table 1. The chemical composition of ash from the main Romanian thermo-power plants

Termocentrala *Thermo-power station*	P.C.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	SiO_2 reactiv *reagent*
	%									
Mintia	1.46-2.29	53.10-53.40	26.5-27.87	8.34-8.84	2.82-3.50	1.51-1.60	0.25-0.38	0.72-0.75	2.22-2.78	38.92-40.97
Doiceşti	1.66-3.78	52.99-55.18	21.20-24.40	8.83-9.75	2.85-4.02	2.31-2.67	0.78-1.80	0.74-0.77	2.67-2.98	39.82-48.11
Rovinari	1.55	48.60	25.50	7.97	8.43	2.39	1.34	0.53	1.72	42.50
Isalnița	2.27-2.33	48.57-49.36	23.18-23.42	8.83-9.10	7.58-8.74	1.90-2.53	1.80-1.93	0.48-0.96	1.77-2.09	38.50-42.54
Oradea	4.35-6.06	44.61-47.57	18.68-19.63	11.18-14.30	7.73-9.25	2.10-2.50	2.62-2.88	0.56-0.79	1.75-1.86	33.43-43.11
Suceava	14.54	45.01	24.88	3.99	5.72	1.79	1.10	0.27	0.93	32.65
Bacău	2.18	51.21	22.27	9.54	6.01	2.39	2.11	0.67	1.97	40.11
Govora	2.73-3.98	47.87-58.41	19.35-20.86	8.81-12.87	4.10-7.86	2.43-3.20	1.38-2.97	0.24-0.34	1.04-1.49	41.32-48.90

Fly ash could be used in concrete production (as a substitute material for Portland cement and sand) or cement clinkers production - (as a substitute material for clay). Fly ash had pozzolanic properties and is used as replacement filler for Portland cement in concrete. Fly ash particles are mixed with water and pumped to an ash impoundment pond. Unfortunately, this deposit over time becomes unstable with the drying of ash and wind action that can carry ash at thousands of miles contaminating farmland or people's homes. At present CET Govora SA is the first and only thermal plant in Romania that has implemented an ash type system as ash for neton grade AN produced for CE certificates, according to SREN 450-1 + A1: 2007 "Type II addition for the production of concrete". At present CET Govora SA is the first and only thermal plant in Romania that has implemented an ash type system as ash for neton grade AN produced for CE certificates, according to SREN 450-1 + A1: 2007 "Type II addition for the production of concrete". Another interest about using of fly ash in Romania comes from LAFARGE Cement România who invested in 9 June 2010, one million euros at Işalniţa thermo-power plants. This fly ash will be used for obtaining special cements.

Kaolin is a clay mineral that has been used to obtain of porcelain. Kaolin could have different colors: white, greyish-white, or slightly colored and formed mainly by decomposition of feldspars (potassium feldspars), granite, and aluminum silicates. Kaolinite is the main structure forming species in the overall geopolymerization process. Davidovits [20] used kaolinite and metakaolin obtained by thermal treatment at 750ºC for 6 hours as source of alumino-silicate oxides to synthesize and produce geopolymers. Other researchers [21, 22] have also focused on the manufacture of geopolymeric products and their industrial applications by using either kaolinite or metakaolinite as the main reactant. Geopolymer could be synthesized by the polycondensation of silico-aluminate structures. Highly alkaline solution of NaOH and KOH could be incorporated with source materials rich in SiO_2 and Al_2O_3 [4] in order to produce geopolymers. Synthesis of geopolymer consists of three basic steps. The first is the dissolution of alumino-silicate under strong alkali solution; this is followed by reorientation of free ion clusters, and the last step is polycondensation [20].

Kaolin in Romania is used for obtaining different types of ceramic materials. Parva kaolin from Romania has been the subject of exploitation since the nineteenth century by private firms, the Nitrogen Society in Târnăveni and the Mica Branch in Bârlad for the ceramic factory in Târnăveni and later by some local enterprises. Kaolin was used in the manufacture of pots and soaps by two factories in Cluj, in 1905 the quantity of 20 tones was extracted. This kaolin was taken to Cluj-Napoca, Romania, being used by the Sanex and Iris ceramic factories, as well as by factories in Comarnic, Alba Iulia and Curtea de Arges or Dorohoi from Romania. It was considered to be a high-grade ore used successfully in the paper industry as well. Today, around Cluj-Napoca there are other kaolin exploitation like S.C. CUART GRUP S.A. Baia Mare, Romania. Another source of kaolin is the holding in SC EXPLOATAREA MINIERA HARGHITA SA [23].

3.5. An overview of the available geopolymer sources in Argentina

Amongst the raw materials used for obtaining geopolymers, fly ash is the most important. It is necessary to find a constant and abundant source of fly ash in order to produce geopolymers in a sustainable way. In that sense, there are two types of fly ash sources: organic and inorganic. The firsts come from the burning process of agricultural by-products like rice husk and wood industries, while the second are residues from coal-fired thermal power plant. In order to produce structural components with geopolymers, the use of fiber reinforcements is recommended. Natural fibers are the best option to reduce the cost and the environmental impact of the resulting materials.

In terms of organic sources for fly ash, Argentina has two regions in which they are abundant, the north province, which is the national center of the wood industry, and the province of Entre Rios where rice is grown. In both cases there are by-products (sawdust and rice husk) that are burned and the energy is harnessed. Currently there are no commercial uses for this resource, and they are mainly disposed in landfills. In the case of fly ash from coal combustion, there is not a massive use of coal as an energy source in the country. However, some thermal power plants are prepared to use both, gasoil and coal alternately. While the first is more common, in the last years the main coal mine in the Patagonia, the Río Turbio coalfield, has been rehabilitated. Therefore, the use of coal is expected to increase and also the availability of fly ash.

The only commercial use for inorganic fly ash currently in the country is as additive for cements. There is only one company that offers the product for the construction industry (Matermix SRL). But the maximum amount of fly ash that can be added to Portland cement is 30%. In the case of geopolymers, Portland would be entirely replaced by fly ashes.

Figure 2. Potential raw materials for project purposes in Argentina

3.6. An overview of the available geopolymer sources in Poland

Development of motorization caused the problem with waste tires. The number of used car tires and other rubber components such as gaskets, tubes - hoses, washers and suspension elements increase year by year. According to estimations, the number of waste car tires it is about 1 billion Mg per year in the world. In Poland, there is produced about 180 000 Mg of tires annually. This mass is reduced by about 20-25% during operation, so it is about 150 000 Mg of waste tires and additionally about to 10 000 Mg of other rubber waste from other parts of vehicles [24,25].

The applied granulate made from rubber (waste tires) was manufactured by company ORZEŁ S.A. Samples were prepared using rubber granules with fractions of 0.0 - 0.8 mm, and rubber granules with fractions of 1 - 4 mm. As the raw material for the production of geopolymers were used fly ash from coal combustion in the grate boilers from the Skawina power plant.

As the sand content increased from 25 to 50%, compression strength increased. The addition of rubber granules negatively affected on the compression strength. The research results show that samples containing rubber granules having a particle size between 1.0 and 4.0 mm have higher compression strengths than the samples with rubber granules having a particle size between 0.0 and 0.8 mm, for the same amount of rubber addition. Comparing the results of series I and II, it can be seen that samples containing 50% fly ash, 25% sand and 25% rubber granulate have slightly lower compressive strengths than the samples containing 75% fly ash and 25% rubber granules (Table 2 and 3).

Table 2. Material compositions and compressive strength after 28 days– I series

Fly ash [% of weight]	The dimensions of granulate	Granulate [% of weight]	Sand [% of weight]	Compressive strength [MPa]
75	–	–	25	32.153
50	–	–	50	33.646
75	0.0 – 0.8	25	–	9.779
50	0.0 – 0.8	50	–	2.189
75	1 – 4	25	–	9.400
50	1 – 4	50	–	2.706

Table 3. Material compositions and compressive strength after 28 days – II series

Fly ash [% of weight]	The dimensions of granulate	Granulate [% of weight]	Sand [% of weight]	Compressive strength [MPa]
75	0.0 – 0.8	12.5	12.5	17.807
50	0.0 – 0.8	25	25	7.454
75	1 – 4	12.5	12.5	17.450
50	1 – 4	25	25	9.960

However the research result for the material with the addition of waste rubber granules show that it have a worse properties than reference material, the using rubber granulate as a filler in fly ash geopolymers have a justification in the conception of sustainable development. Despite the negative impact of the rubber granulate on the material properties such a compressive strength of the material obtained, using it as a filler in geopolymers gives clear environmental benefits, including the possibility to recycling waste rubber and reduce the consumption of natural materials.

4. Conclusions

This research report presents the result for the studies performed by the project members for identifying suitable waste products for new composite materials based on geopolymer matrix reinforced with waste fibres. The main waste material candidates for each country are:

- Peru – recycled clay bricks and natural pozzolana from volcanic ash;
- Turkey – fly ash resulted from burning fossil fuels in large scale power plants (Çatalağzı Termal Power Plant and Isken Sugözü Termal Power Plant);
- Uruguay – Paper mill ash from paper mills and rice hull ash from power generation plants;
- Romania – fly ash resulted from coal burning in thermal power plants;
- Argentina – fly ash resulted from the burning of organic waste materials (sawdust and husk rice) as well as fly ash resulted from the burning of fossil fuels in power plants;
- Poland – granulated rubber resulted from waste tyres

5. Bibliography

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6. Mobilities

Three mobilites were conducted during 2017 for the ERA-Net LAC FIBRE project.

a) First project meeting

The first project meeting was held in Krakow, Poland, representatives for each project partner being present. Each member of the project held a presentation with the introduction of the research team, a brief presentation of their research facilities and their role in the project.

The second part of the meeting consisted of a conference, which aimed the outlining of the first Work Project. The project members participating in WP1 presented their preliminary studies and their objectives. The proceedings of the conference were subsequently published.

A visit of the research facilities of the Technical University of Krakow as was also programmed.

b) Technical University of Munich visit

The main goal of the TU Munich mobility was the Materials Science and Mechanics of Materials laboratory visit, for a potential collaboration regarding the testing and simulation of the materials developed in the project. Staff exchange, as part of a bilateral agreement, was also considered.

c) Second project meeting

The second project meeting, held in Krakow, aimed the dissemination of the partial results of the first Work Package. The Krakow team supplied the UPT team with materials manufactured as part of WP1, and an experimental plan was developed (for WP3 and WP4).

Another discussed topic was the organization of the third project meeting, which will be held in Timișoara, Romania. The meeting agenda was detailed, for each planned activity.