

OSMAN GENCEL¹⁾, WITOLD BROSTOW^{2),*)}, GONZALO MARTÍNEZ-BARRERA³⁾,
MUSTAFA SABRI GOK⁴⁾

Mechanical properties of polymer concretes containing different amount of hematite or colemanite

Summary — Polymer concretes (PCs) were created by using varying concentrations of silica sand as aggregate and also hematite, colemanite and a commercial epoxy resin. Hematite is known to exhibit radiation shielding properties. Mechanical performance was evaluated including unit weight, pulse velocity, compressive strength, flexural strength, splitting tensile strength, modulus of elasticity and dynamic elastic modulus. As expected, mechanical properties depend on the resin concentration, the aggregate type and aggregate content in the mixture. Hematite particles have a larger effect than colemanite on mechanical properties of PCs. Inclusion of hematite provides significant improvement of the mechanical properties studied PCs in comparison to PC with silica sand only.

Keywords: polymer concrete, hematite, colemanite, epoxy resin, mechanical properties.

WŁAŚCIWOŚCI MECHANICZNE BETONÓW POLIMEROWYCH ZAWIERAJĄCYCH RÓŻNE ILOŚCI HEMATYTU LUB KOLEMANITU

Streszczenie — Wytworzono próbki betonu polimerowego (PC) na bazie żywicy epoksydowej napełnionej różną ilością kruszywa, którym był piasek kwarcowy oraz kolemanit lub hematyt. Hematyt zastosowano ze względu na jego zdolności ochronne przed radiacją. Badano gęstość i właściwości mechaniczne otrzymanych betonów, w tym: prędkość rozchodzenia się fal ultradźwiękowych, wytrzymałość na ściskanie, wytrzymałość na zginanie, wytrzymałość na rozciąganie, moduł sprężystości oraz dynamiczny moduł sprężystości. Zgodnie z oczekiwaniami, właściwości mechaniczne zależą od zawartości żywicy w betonie oraz rodzaju i ilości kruszywa w mieszance. Większy wpływ na właściwości mechaniczne mają cząstki hematytu niż kolemanitu. Dodatek hematytu zapewnia znaczną poprawę badanych właściwości mechanicznych betonów w porównaniu z właściwościami PC napełnionego wyłącznie piaskiem kwarcowym.

Słowa kluczowe: beton polimerowy, hematyt, kolemanit, żywica epoksydowa, właściwości mechaniczne.

Concretes are the most widely used construction materials in the world; a large variety of concretes is based on a variety of cements [1]. Low costs, ease of application and high compressive strength are the main factors to be considered for a given application. Inorganic (mineral) con-

cretes based the Portland cement have shortcomings: poor flexural strength, low tensile strength, high porosity, freeze thaw deterioration, destruction by corrosive chemicals *etc.* [2, 3]. These shortcomings are viewed as more and more acute since we have become more and more concerned with conservation of energy and materials.

One approach consists in various property improvement staying within the range of mineral concretes, for instance using polymeric fibers as a reinforcement [4, 5]. Another approach is based on a combination of technologies of concrete and that of polymers [6]. Thus, polymer concrete (PC) materials have become a viable choice for the civil construction sector in developed countries, particularly in applications such as making reinforced slabs, overlays for highway pavements and bridge decks or pipe coatings. PCs are also used in repairing deteriorated mineral concretes (Portland cement concrete) in situations when high strength, fast cure and durability are required [1]. Polymer concretes are composites in which the

¹⁾ Bartın University, Faculty of Engineering, Civil Engineering Department, 74100 Bartın, Turkey.

²⁾ University of North Texas, Department of Materials Science and Engineering and Center for Advanced Research and Technology, Laboratory of Advanced Polymers and Optimized Materials, 1150 Union Circle # 305310, Denton TX 76203-5017, USA.

³⁾ Universidad Autónoma del Estado de México, Facultad de Química, Laboratorio de Investigación y Desarrollo de Materiales Avanzados (LIDMA), Km.12 de la carretera Toluca-Atzacomulco, San Cayetano 50200, Mexico.

⁴⁾ Bartın University, Faculty of Engineering, Department of Metallurgy and Material Engineering, 74100 Bartın, Turkey.

*) Corresponding author; wbrostow@yahoo.com

aggregates are bound together in a polymer matrix [7]. They do not contain Portland cement. Czarnecki [8] claims that in the next 25 years it will be necessary to build the same number of dwellings as existing today in order to meet demands for new housing. The need to double the number of buildings on the Earth's surface drives specialists to analyze the past performance of the civil construction sector and reflect on possible actions to deal with the issues that have to be addressed. The use of new materials may contribute to creation of a new improved scenario in this sector. According to his data, the annual production of cement is approximately $1.2 \cdot 10^9$ tons, yielding approximately $4 \cdot 10^9 \text{ m}^3$ of concrete. If only a small fraction of this is used to produce polymer concrete compounds, a good market for these materials results.

Another area in where polymer concretes are needed is radiation shielding. For shield design, neutrons and gamma rays are the main types of nuclear radiation to be considered. Since any shield that attenuates neutrons and gamma rays will be effective for attenuating other radiations in facilities such as nuclear power plants, particle accelerators, research reactors, laboratory hot cells and medical facilities where radiation impermeability is required. Shielding is designed to combine the most effective shielding components into a single homogeneous composite — while satisfying specific shielding requirements for radiation sources. Recently, there is a continuous demand for improved polymers that satisfy stringent requirements such as high mechanical strength, adhesiveness and heat resistance for the use as shielding materials. These requirements, which often involve a combination of many properties difficult to attain, could be satisfied by utilizing composites which can act synergistically to solve application needs. Fillers played the key role in satisfying such requirements. Since polymeric materials are on their own hydrocarbonic substances, we would expect good neutron moderation. With judicious choice of heavy mineral or metal fillers, gamma rays and X-rays could be also shielded. Therefore, the hematite mineral (which is well known for its high attenuating properties) was incorporated as an aggregate to obtain epoxy-hematite concretes developed for biological concrete shields as well as for neutrons and gamma rays attenuation [9]. To provide sufficient shielding, effects of irradiation on polymers need to be known; some literature on this subject exists [10–14].

Recently we see upgrades of radiation sources or machinery in existing facilities. Generally, the radiation strength becomes stronger when machinery is upgraded. Only safe machines that decrease radiation leakage are designed. However, in many cases, this is not enough. There are two methods used to achieve additional shielding. One is increasing thickness of concrete shielding on the wall surfaces — what results in the room becoming smaller. The other option consists is adding a self-shielding cover around the machinery [15].

Bearing these situations in mind, this study focuses on mechanical properties of polymer concretes containing different hematite and colemanite proportions for use as structural components in civil engineering applications, and especially as new radiation shielding materials.

EXPERIMENTAL

Materials

Polymer concretes are produced by using dry aggregates and monomers (binders) that undergo polymerization (curing, hardening). We have used epoxy resins which have a large variety of applications [16–21]. They exhibit good dimensional stability, high heat resistance, high mechanical strength and chemical resistance. They are used as floor painting in radiation facilities and for nuclear fuel casks because of their relatively high resistance to gamma rays. The selection of the type of epoxy resin was based on the requirement of curing at room temperature, heat evolved during curing on the low side and pot-life suitable for industrial applications. As a result, while the hardener is an aliphatic polyamine, an epoxy resin of the diglycidyl ether of Bisphenol-A type used as an epoxy resin was purchased from System Three Resins (Auburn, Alabama). Its epoxide equivalent weight, viscosity at 25 °C and density are 210 g/eq, $1.10 \cdot 10^3 \text{ cP}$ and 1.10 g/cm^3 , respectively. Those chemicals were used as received.

Medium-size river sand with a regular particle size distribution and density 2.65 g/cm^3 was used. Particle distribution of sand is provided in Table 1.

Table 1. Gradation of mineral particles used to obtain polymer concretes

Aggregate	Sieve size						
	2.36 mm	1.18 mm	600 μm	425 μm	180 μm	150 μm	75 μm
percentage passing							
Hematite	100	90.91	79.39	71.52	49.09	38.99	16.36
Colemanite	100	94.97	88.93	83.70	62.37	51.11	25.96
Sand	100	92.87	83.45	75.68	56.61	46.05	20.56

Colemanite ($2 \text{ CaO} \cdot 3 \text{ B}_2\text{O}_3 \cdot 5 \text{ H}_2\text{O}$) is a calcium borate mineral with hardness between 4 and 4.5 and density of about 2.4 g/cm^3 [22]. Pure colemanite has a B_2O_3 content of about 51 wt. %. Turkey has an abundance of boron minerals, namely about 60 % of the world reserves. Research on neutron shielding materials using colemanite has been going on for more than 40 years. The aim was to make concrete using colemanite [15]; however, our previous study [23] has shown that incorporating colemanite into concrete worsens physical and mechanical properties of concrete. The reason is

that colemanite has poor rock strength and is soluble in water. Therefore, it is difficult to make concrete which maintains its quality; however, concrete blocks containing colemanite can be made [24]. Chemical composition of colemanite and gradation of particles are presented in Tables 1 and 2, respectively.

Table 2. Chemical composition of colemanite

Component	Content, wt. %
B ₂ O ₃	41.24
CaO	24.35
MgO	1.42
Fe ₂ O ₃	0.44
SiO ₂	5.07
LOI ^{a)}	24.28

^{a)} Loss of ignition.

Hematite is a natural red rock that contains iron oxide, has a Mohs hardness between 5.5 and 6.5 and the density between 4.9 and 5.5 g/cm³. However, physical properties of rocks in which hematite are the main constituent may vary considerably; the density of hematite ores can range between 3.2 and 4.3 g/cm³. Although some ores are soft and produce dust in the course of being handled, which would make them a poor aggregate for heavy concrete, and also hematite particles tend to be flaky, which is undesirable in regard to the workability of concrete [22], our previous study [25] shows that hematite added concretes with high physical and mechanical properties can be produced by a good mixture design and technique. Besides, another study by some of us [26] has shown that concrete containing hematite can be used as a biological shield against ionizing radiation. In that work, in order to measure absorption properties of concrete using colemanite and hematite on rats, a cage model was used. Rats were housed in concrete cages and irradiated with 7 Gy gamma rays from LINAC (Siemens, Erlangen, Germany) twice a week. The extent of gamma-irradiation-induced liver damage and oxidative stress in rats were evaluated and found low.

Table 3. Chemical composition of hematite

Component	Content, wt. %
Fe ₂ O ₃	82.26
MnO	0.13
MgO	1.54
TiO ₂	0.03
Al ₂ O ₃	0.57
CaO	4.68
SiO ₂	4.15
LOI	5.63

Thus, although hematite has been successfully used for shielding in concrete, we have not found in the literature information about polymeric resin-hematite composites. Hematite was prepared as aggregate by crushing and grinding the ore in a laboratory mill, then sorting it *via* sieves. The chemical composition of hematite is presented in Table 3.

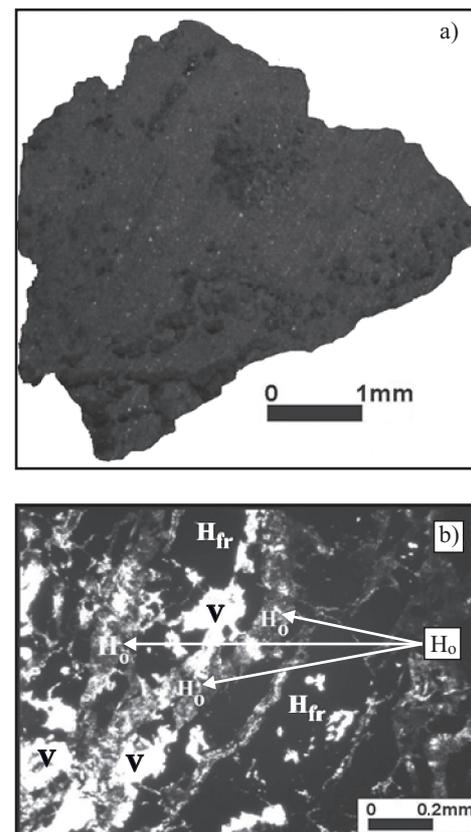


Fig. 1. Mineralogical structure of hematite: a) general view of aggregate, b) structure of aggregate with oxide zones (H_O), fresh zones (H_{fr}) and voids (V)

Mineralogical structure of hematite is presented in Figure 1. We see in that figure that hematite consists of two zones, oxide (H_O) and fresh (H_{fr}). Hematite aggregates have high porosity (Fig. 1b) — a consequence of voids (V) which have appeared during formation of hematite. We also note that hematite has the form of flakes. In this study, we kept the aggregates in an oven to avoid moisture for 4 h at the temperature of 105 ± 5 °C.

Polymer concrete compositions

Since polymer concrete is a heterogeneous material, the properties may be quite variable. Contributions to the variability include heterogeneity of the aggregate particles and polydispersion of the polymer binder.

Different types, properties and applications of polymer concretes have been extensively reported [7, 27, 28].

However, there is limited information for PCs on material heterogeneity, materials properties and their relations to the kind of aggregate (mineral type, grain grade-natural and crushed) as well as to the maximal grain diameter. Thus, more knowledge of the optimal compositions and mechanical properties including stress-strain relationships *etc.* are necessary to enable appropriate design, production, and quality control [29].

Applications and performance of polymer concretes are dependent upon the specific polymer binder as well as the type of aggregate and its gradation [3]. Thus, the choice of the fine aggregates is very important in order to achieve the best workability of the PCs. Moreover, the mix design of PCs typically involves an aggregate gradation to provide the lowest possible void volume. Thus, a minimum polymeric binder contents necessary to coat the aggregates and to fill the voids has to be defined [29]. The distribution of aggregates should be such as to allow for a minimum void volume for dry packed aggregates — resulting in dense packing. Dense packing of aggregates in the polymer concrete matrix is known to result in better properties [30].

Table 4. Compositions of the polymer concrete specimens

Specimen codes	Epoxy resin wt. %	Sand wt. %	Hematite wt. %	Colemanite wt. %
R	20	80	—	—
H1	23	61.6	15.4	—
H2	27	36.5	36.5	—
H3	32	17	51	—
H4	40	—	60	—
C1	23	61.6	—	15.4
C2	27	36.5	—	36.5
C3	32	17	—	51
C4	40	—	—	60

In the present study, polymer concretes containing hematite and colemanite based on an epoxy resin were optimized and reported. The compositions used are summarized in Table 4.

Methods of testing

We have determined compressive strength, flexural strength, splitting tensile strength and elasticity modulae for a variety of compositions we have prepared.

A mixing procedure according to the ASTM C305 was followed. A laboratory type Hobart mixer was used. First, aggregates were put in the mixer bowl, and then the mixer was started. While the mixer was running, the epoxy resin was added. The operation consists of two stages: mixing for 1.0 min at the paddle speed of 140 rpm, followed by 1.5 min at the speed of 285 rpm. After mixing, cubic specimens with the side of 50 mm for compres-

sion testing, cylindrical specimens with 76 mm in diameter and 152 mm in length for splitting tensile and modules of elasticity tests, prismatic specimens with dimensions of 50 × 50 × 305 mm for flexural test were fabricated. After casting, the molds were subjected to vibration on a vibration table. Thus, the air was evacuated from the samples. PC specimens were kept at 23.0 ± 3.0 °C for 7 days.

The axial compressive strength test was performed according to the ASTM C 109M standard. The specimens were tested in a hydraulic load machine at a constant loading rate of 120 kg/s. The beams were loaded in third-point loading at a uniform rate of 225 kg/min; the secant modulus of elasticity was determined. The compression elastic modulus was calculated at the stress 40 % of the max maximum strain on the stress-strain graph. Testing of the specimens was performed at 7 days.

RESULTS AND DISCUSSION

Density

The density (unit weight) of prepared concretes are presented in Figure 2.

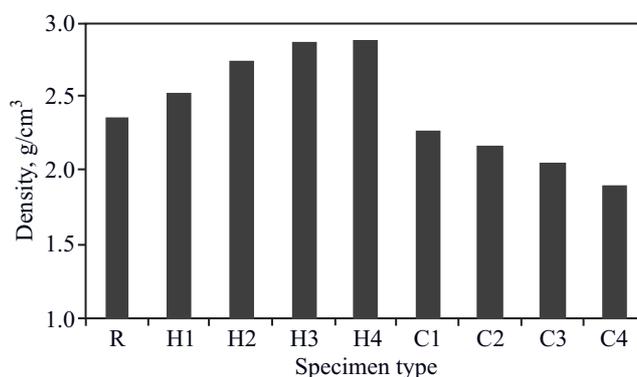


Fig. 2. Effect of PC specimens composition on their unit weight

As seen clearly from the figure, the density increases with increasing hematite content while it decreases with increasing colemanite content. The reason for this are density values for hematite and colemanite. Since hematite has higher density than sand, addition of hematite increases the density of concrete — an expected but also a desired result. Values of density ranges from 1.9 to 2.8 g/cm³. The maximum value, 2.8 g/cm³, is obtained for the material H4 whereas the minimum, 1.9 g/cm³, is for C4.

The probability of an incoming photon interacting with a given material per unit path length is usually represented by the linear attenuation (also called linear attenuation coefficient) — clearly pertinent for radiation shielding. Needless to say, the attenuation depends on the density of the material [14, 31]. Thus, density value of concretes is important. The higher the density, the smaller

the thickness of concrete is required to provide radiation shielding.

Pulse velocity

Ultrasound testing is a well known non-destructive method and we have used it for determination of several properties. The results of ultrasound pulse velocities tests are presented in Figure 3.

Figure shows pulse velocity values ranging from 4.0

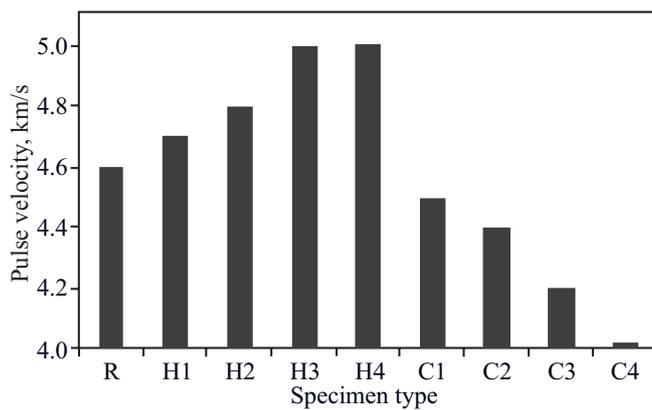


Fig. 3. Effect of PC specimens composition on their ultrasound pulse velocity

to 5.1 km/s. The velocity increases with increasing hematite concentration and the highest value is for the H4 material. Increasing colemanite concentration lowers the velocity. These results can be explained by the well-known fact that sound travels faster in a more compact medium. This is why Figures 2 and 3 have similar shapes.

Compressive strength

There is no need to argue that the compressive strength is the most important property of concrete and

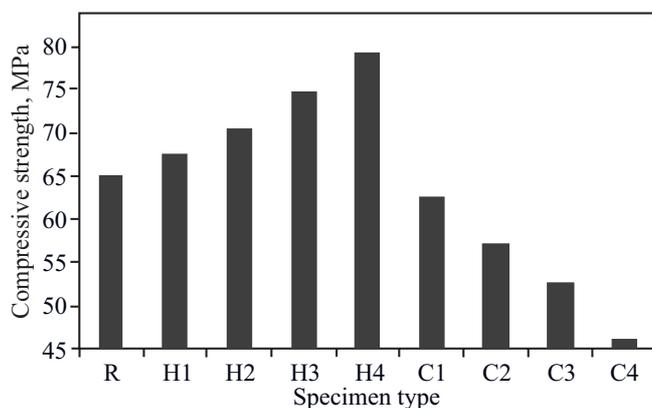


Fig. 4. Effect of PC specimens composition on their compressive strength

an indicator of the overall performance. The results are shown in Figure 4.

In general, the compressive strength increases when the hematite content increases. Thus, the values for hematite containing concretes vary from 67.6 to 79.1 MPa, an improvement up to 21 % for this class of concretes. For concretes containing colemanite, we find a decrease from 62.7 to 46.2 MPa when increasing the colemanite concentration, a significant effect. We note however that resin + colemanite compositions are in use in facilities such as boron neutron capture therapy rooms where light and effective neutron shielding materials are needed. When available, C4 can be used as bricks or slabs to cover walls and/or pavements.

Flexural strength

The results of flexural strength determinations are presented in Figure 5.

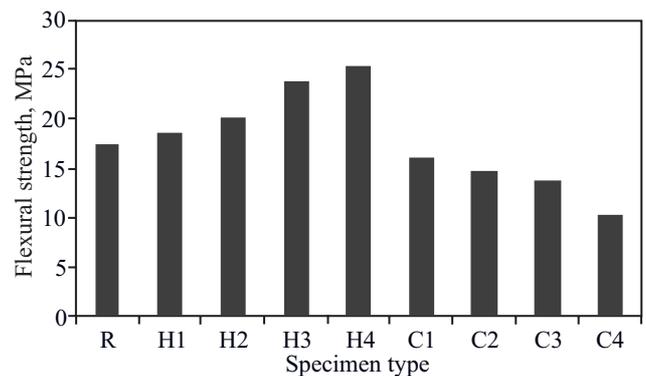


Fig. 5. Effect of PC specimens composition on their flexural strength

We find that the flexural strength values go symbiotically with the hematite and resin content. The values for hematite concretes vary from 16.6 to 25.3 MPa. In the colemanite series we find a decrease from 16.2 to 10.2 MPa, even though the resin content increases. These results can be explained in similar terms as those in earlier figures. A factor playing a role can also be the strength of the bonding between the particles and the epoxy resin. Kopczyńska and Ehrenstein stress the role of interfaces for properties of multiphase composites containing polymers [32] — a role also seen in our earlier papers [33]. The bond between coarse aggregate and cement paste is known to be the weakest region in ordinary concrete. This bond fails first upon loading, initiating fracture of the concrete. The weakness of the bond is attributed to the presence between the aggregate and cement paste of an interfacial transition zone, which is a porous calcium hydroxide-rich layer [34–36]. From our results, a plausible interpretation is that hematite particles exhibit bonding to the resin. The tensile strength of concrete

is in some cases assumed to be zero in concrete design. However, in certain structures the tensile strength must be known. This is the case for dams, airfield runways, roads, pavements and other slabs [37]. In this respect, we can see the advantages of using hematite as the aggregate. Moreover, a further improvement can be achieved by inclusion of polypropylene fibers and gamma irradiation [38, 39].

Splitting tensile strength

The results of splitting tensile strength measurements are shown in Figure 6.

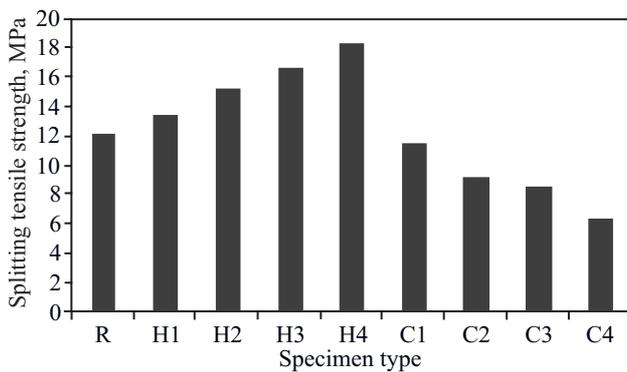


Fig. 6. Effect of PC specimens composition on their splitting tensile strength

Also here we find that PCs containing hematite show a increasing trend — contrary to PCs containing colemanite. Aggregate quality and matrix bond is important for the splitting tensile strength. Splitting strength values of PCs with colemanite are significantly reduced because more weak points appear — due to weak adhesion at mortar-colemanite interfaces. Further, load bearing capacity of colemanite particles is quite low. Return now briefly to mineral concretes discussed in the beginning of this article. Even for colemanite containing samples, the values are three to five times higher than for mineral concretes.

Compression modulus of elasticity

The results modulus of elasticity determination are illustrated in Figure 7.

The modulae seen in Figure 7 are known to be related to the compressive strength and density. Another pertinent factor is that the aggregate is important for volume stability but it also affects the strength and the elastic modulus of concrete.

The elasticity modulae in Figure 7 exhibit similar behavior as the compressive strength results. The values range from 35.4 to 42.3 GPa for the hematite series and from 32.8 to 24.2 GPa for the colemanite series. The values for the former are thus above that for the reference sample for the former and below that value for the latter. We

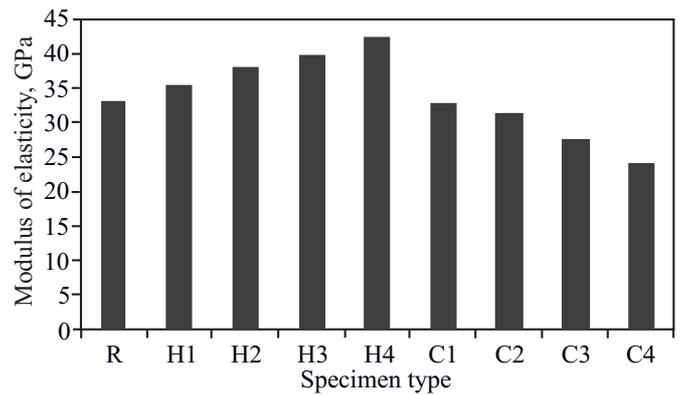


Fig. 7. Effect of PC specimens composition on their comparison modulus of elasticity

see the highest value for H4, namely 42.3 GPa. Let us compare the present values to those for PCs elaborated with different mineral aggregates. Our values — for both hematite and colemanite series — are higher than for a PC with silica sand + CaCO₃ [1], for PCs with two mineral aggregates, marble/calcium bentonite or for PCs with one aggregate: silica sand [40] or CaCO₃ [41]. Apparently, more ductility is achieved when using hematite particles instead of silica sand.

Dynamic elastic modulus

Ideally, this modulus is measured directly on concrete samples under compression by recording the load-deformation curve. However, this is not always easy. To avoid demanding and time-consuming direct measurements of elastic modulus (E_c), simplified approaches with either a theoretical or an empirical basis have been developed [1].

Non-destructive tests take into account the acoustic impedance of the system components — important factors influencing ultrasonic wave propagation [42]. The dynamic elastic modulus is determined by measuring the pulse velocity along the composite and using electrical transducers located on the opposite sides of the cubic specimens of concrete. The energy supplied by the ultrasound depends on how compact is the composite, including the void presence and the void sizes. One thus obtains the dynamic elastic modulus E_d :

$$E_d = U^2 \rho (1 + \mu) (1 - 2\mu) / (1 - \mu) \quad (1)$$

where: U — the pulse velocity, ρ — the mass density, μ — the Poisson ratio.

E_d necessarily depends on the component properties and their interactions with the matrix.

Dynamic elasticity modules of concretes are presented in Figure 8.

The dynamic elasticity modulus exhibits similar behavior as the compression modulus of elasticity seen above. However, the dynamic values are higher than the static ones, a consequence of not using mechanical loads when determining the latter [36, 43]. Our values range from

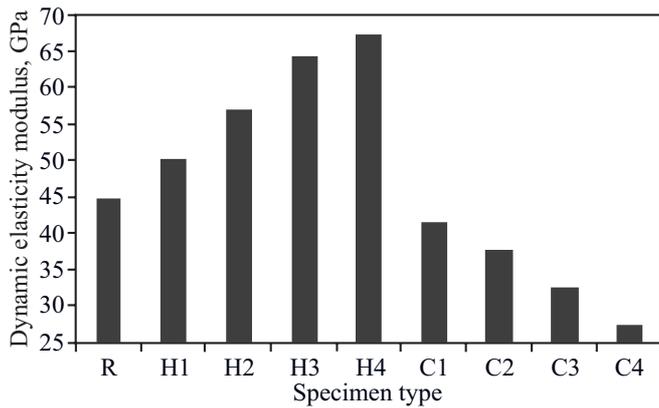


Fig. 8. Effect of PC specimens composition on their dynamic elasticity modulus

50.1 to 67.4 GPa for PCs with hematite and from 41.4 to 27.4 GPa for PCs with colemanite. When compared to R sample (with silica sand only), an increase in the E_d values are found for all concretes containing hematite up to 50 % and a decrease for PCs with colemanite by 40 %.

A SURVEY OF RESULTS

We have investigated the influence of hematite and colemanite on mechanical properties of polymer concretes. There is an increase in axial compressive strength as the concentration of hematite increases. Conversely, there is a decrease in compressive strength as the concentrations of colemanite increases.

A high modulus of elasticity was obtained for the hematite sample containing the highest hematite content, with the peak value equal to 42.3 GPa. Modulus of elasticity values of concretes with colemanite decreased by depending on colemanite content. However, all composition presented fairly high ductility.

Hematite increased unit weight of concretes whereas colemanite decreased dependent on concentration. Effects of the hematite particles presence on mechanical properties of polymer concretes is more pronounced than that of colemanite. Finally, we note that in general polymer-based composites can achieve better properties than mineral concretes [44].

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POLIMERY – NAUKA – PRZEMYSŁ 2012
Częstochowa — Słok k. Bełchatowa, 17–19 września 2012 r.

Przewodnicząca Komitetu Naukowego: prof. dr hab. inż. Elżbieta Bociąga

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