Enhancing fire resistance of concrete through metakaolin substitution: A comprehensive experimental study

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Abstract
In the pursuit of sustainable construction practices, the use of eco-friendly materials in concrete production has garnered considerable interest. This study meticulously analyzes and compares conventional concrete with concrete incorporating metakaolin as a cement substitute, focusing on its impact on fire resistance properties. It examines mechanisms such as metakaolin's influence on other mechanical properties. A systematic experimental approach assesses the fire performance of concrete specimens with varying metakaolin proportions (5%, 10%, and 20%). These specimens were tested for pre- and post-fire compressive strength, flexural strength, porosity, density, and water absorption. Results indicated that 5% metakaolin provided comparable fire resistance and compressive strength to conventional concrete, although density decreased while porosity increases with higher metakaolin content. The study also addresses the practical implications of using additive-enhanced concrete in real-world construction. This research underscores the importance of environmentally friendly construction solutions while ensuring concrete's durability and safety under fire conditions, contributing to the understanding of sustainable concrete materials and their fire-resistant performance.
Keywords: Metakaolin, Fracture Energy, Elevated temperature, Flexure tensile strength
Kulcsszavak: metakaolin, törési energia, magas hőmérséklet, hajlító-húzószilárdság

1. Introduction

Calibrated kaolinite clay is the source of metakaolin, a pozzolanic substance that is distinguished by its composition of thermally activated alumino silicates. As a primary product as opposed to a secondary or by-product, metakaolin stands out from typical pozzolans. The concrete has a lighter color when metakaolin is present. By accelerating the hydration of ordinary Portland cement (OPC), filling the void, and initiating a pozzolanic reaction with calcium hydroxide ($Ca(OH)_2$), metakaolin strengthens and prolongs the life of concrete [1, 2]. It is acknowledged that the principal factor causing the susceptibility seen at the interface between cementitious materials and aggregate particles is calcium hydroxide ($Ca(OH)_2$). It consequently has a major effect on the properties of concrete that are connected to durability, porosity, permeability, and strength [3, 4]. Concrete loses some of its qualities when it is exposed to high temperatures, like during a fire. This involves serious problems such as decreased compressive strength, spalling and fissures, harm to the link between aggregates and cement paste, and slow degradation of the hardened cement paste. Pozzolanic materials can be used to replace regular Portland cement, improving the cement's mechanical and fire resistance qualities [5]. However, it is possible to lower down the negative effects of temperature on concrete, nevertheless, by taking preventative steps, such as choosing the right materials. The monitoring and prediction of concrete behavior is greatly aided by an understanding of the material properties, including those of cement paste, aggregate, the link between aggregate and cement paste, and the thermal compatibility of their combination [6]. Concrete's ability to withstand fire is largely influenced by the materials that are used. Concrete's ability to withstand fire is discovered to be significantly influenced by the pozzolans that are typically added to it to increase its strength and endurance. To counteract the structural degradation of concrete, researchers have investigated using other binders or adding cement substitutes in part. Materials like silica fume, metakaolin and other supplemental cementitious materials are currently showing promise as feasible solutions that satisfy a range of performance, cost, and environmental requirements [7, 8]. The temperature range that is required to calcine metakaolin usually ranges from 550 °C to 700 °C, which has benefits for sustainability. In comparison to the energy and temperature requirements for clinker manufacturing, which normally calls for temperatures of over 1400 °C, this temperature range is much lower [9]. This suggests that the manufacture of metakaolin requires less energy and money than the production of cement, acknowledging that regular Portland cement is a significant source of carbon dioxide emissions. Furthermore, metakaolin enhances the performance of construction projects by means of its filler effect and pozzolanic chemical reaction with $Ca(OH)_2$. Among other advantages, this improvement includes greater resistance to sulfate, decreased shrinkage and creep, decreased chloride infiltration, and better durability and service life [10]. This paper conducts a thorough review of existing literature on the impact of metakaolin (MK) on heated concrete properties. It also examines current carbon dioxide (CO_2) emissions in the cement industry and explores how metakaolin could potentially reduce CO_2 emissions and promote environmentally friendly building practices. The substitution of metakaolin (MK) for cement in concrete has been found to enhance mechanical and durability properties, particularly with an optimized replacement ratio. The effectiveness of this ratio is influenced by the characteristics and fineness of the metakaolin. For this study, four concrete mixes containing 0%, 5%, 10%, and 20% metakaolin were prepared and then subjected to high temperatures for analysis.

2. Experimental Study

2.1 Materials

The cementitious material used in all mixtures included ordinary Portland cement Cem I (52.5 N), in compliance with EN 197-1:2011 standards, and metakaolin (Metaver) meeting the NF EN 206-1/CN specifications. To achieve the adequate consistency superplasticizer MasterGlenium 300 was added in varying dosages across the four mixes. *Table 1*, based on the manufacturer's data, provides a comprehensive overview of the chemical composition and the physical and mechanical properties. The fine aggregate used in the mixtures was natural sand, and natural quartz gravel was utilized as the coarse aggregate, both conforming to the standards specified in EN 12620:2002+A1:2008. *Table 2* provides detailed information on particle size, mixing ratios of the aggregates. Standard potable tap water, in accordance with EN 1008:2002, was used for producing and curing the concrete specimens.

2.2 Mix proportions

Four concrete mixes were developed, each with a water/cement (w/c) ratio of 0.45 and 390 kilograms of cementitious material per cubic meter. These included a control mix with 0% metakaolin and three other mixes incorporating metakaolin at 5%, 10%, and 20%, respectively. *Table 3* details the specific proportions of each concrete blend with varying metakaolin content.

Component (%)	Cement (CEM I, 52.5 N)	Metakaolin (MK)
SiO_2	19.73	51.8
Fe_2O_3	3.21	45.8
Al_2O_3	5.55	0.35
CaO	65.02	0.01
MgO	1.44	0.03
SO_3	2.88	-
Na_2O	-	0.13
K_2O	0.78	0.06
Cl	0.0048	-
Specific surface area	4500	22000
Colour	Grey	white

Table 1 Properties of cement (Cem) and Metakaolin (MK)
1. táblázat Cement (Cem) és metakaolin (MK) tulajdonságai

	Fine aggregate	Coarse aggregate
	Natural Sand	Natural Aggregate
Particle size (mm)	0/4	4/8
Mixing ratio (%)	43	57

Table 2 The particle size, Mixing ratio, of aggregates
2. táblázat Adalékanyag szemcseméret, keverési arány

2.3 Mixing, casting, curing, heating, and cooling details

Concrete incorporating Metakaolin (MK) was mixed, poured, and compacted according to EN 12350-5:2019 standards. Mixing was done using a power-driven rotating pan mixer, and samples were cast and compacted using a vibrating table. Fresh concrete was poured into molds and left to set for 24 hours in the lab. After demolding, specimens were submerged in a water tank at 23 ± 1 °C for a week. After one week of water tank curing, they were conditioned in the lab for about 21 days. The test specimens were dried before the heat load to avoid the spalling effect. They were dried at 60 °C for four days. Specimens were then heated in an electric muffle furnace to target temperatures of 20, 100, 200, 300, 400, 500, 600, and 800 °C. Heating started at room temperature, with the temperature rising at a rate of 5 °C to 6 °C per minute until reaching the target temperatures. Specimens were held at these temperatures for 2 hours under steady-state conditions. After heating, the furnace was turned off, and specimens were allowed to cool slowly in ambient air for 24 hours.

2.4 Testing procedure and methods

The Compressive strength (*fc*), Flexure strength (*fl*), Water absorption (*Wa*), Density (*ρ*), and Porosity (*P*) and Fracture energy (*Gf*) were evaluated for control concrete (0%) and concrete with 5%, 10%, and 20% metakaolin. The fc and fl tests were conducted according to EN 12390-3:2019 Part 3 and EN 12390-5:2019 Part 5 on cubic specimens (15 cm sides) at 14, 28, and 90 days, and on prisms (27 x 7 x 7 cm) at an average age of 40 days. Post-fire compression tests (fcp) were carried out on half prisms (7 cm sides) after exposure to temperatures ranging from 20 to 800 °C at an average age of 46 days. The compressive strength (fc) was measured using a 3000 kN capacity compression machine at a loading rate of 0.6 MPa/s. Flexural tensile strength was determined with a flexural tensile testing machine, adhering to EN 12390-5:2019 Part 5 standards. Water absorption (Wa) tests were performed on 7 x 7 x 7 cm concrete cubes following EN 1936:2007. Twelve specimens per mix were tested at an average age of 40 days. The water absorption was calculated by immersing dried samples (Msd) in water at 20 ± 2 °C until constant mass (Ms) was achieved. The water content (Wc) was derived from the difference between the saturated (Ms) and dried (Msd) masses. The water absorption percentage (Wa) was calculated using the appropriate equation.

$$Wa\ (\%) = \frac{(Ms - Msd)}{(Msd)} \times 100 \qquad (1)$$

Density (ρ) and porosity (P) are crucial factors influencing concrete's functionality and structure. In this study ASTM

C1754 procedures was followed to assess (ρ) and (P). Samples were subjected to a 60 °C oven for 4 days to determine density, with their hardened weight recorded. Apparent porosity (P) was evaluated by submerging samples in water for 24 hours to obtain submerged weight. Calculation of apparent porosity (P) involved using submerged weight, oven-dried weight, volume of water absorbed (Vw), and total specimen volume (V), in accordance with ASTM C1754 guidelines.

$$(P)apparent = \frac{(Vw)}{(V)} \quad (2)$$

The fracture energy (*Gf*) was conducted according to the guidelines published in the JCI-S-001-2003.

Mix	Percentage	Cement (kg/m³)	MK (kg/m³)	Water (kg/m³)	NS (kg/m³)	NA (kg/m³)	SP (kg/m³)
0 MK	0%	390.0	0	175.5	782.4	1037.1	0.221
5 MK	5%	370.5	19.5	175.5	782.4	1037.1	0.395
10 MK	10%	351.0	39.0	175.5	782.4	1037.1	0.207
20 MK	20%	312.0	78.0	175.5	782.4	1037.1	0.273

MK = metakaolin, NS = natural sand, NA = natural aggregate, SP = superplasticizer

Table 3 Mix proportion of concrete containing metakaolin (Concrete mix designs for 1 m³)
3 táblázat Metakaolin tartalmú betonok keverési arányai (beton receptúrák 1 m³-hez)

3. Test results and discussion

Various plots were created to identify and analyze the physical and mechanical properties of concrete, thereby evaluating the effectiveness of metakaolin.

3.1 Apparent Porosity and Density

Apparent porosity (P) is critical for concrete's strength and durability. To measure it, samples were oven-dried, weighed, submerged in water, and re-weighed periodically until saturation. Using dry, saturated, and submerged weights, porosity was calculated. *Fig. 1* shows that increasing metakaolin dosage generally increases porosity, likely due to rapid pozzolanic reactions with $Ca(OH)_2$, producing additional hydration products. Variations in curing conditions and the water-cement ratio also influence porosity. Over time, entrapped water in the concrete matrix is consumed in the pozzolanic reaction, enhancing strength. [11, 12] Study and It was observed that when the metakaolin content was less than 20%, there was a decrease in the overall porosity of the paste. However, when the metakaolin content exceeded 30%, porosity increased, possibly due to the 'filler effect' of fine metakaolin particles and the higher water-to-binder (w/b) ratios associated with increasing metakaolin content. After 100 days of curing, the inclusion of metakaolin led to a reduction in both the pore volume of the mortar and the threshold diameter [13]. Suggests the analysis of the porosity and pore size distribution of cured OPC-metakaolin paste indicates that the incorporation of metakaolin refined the pore structure of the cement paste. Moreover, an increase in metakaolin content resulted in a decrease in the threshold pore size [13]. Also noted that with the increase in the water-to-binder ratio (w/b), the porosity of a paste increases. Hence, it can be deduced that Metakaolin has a more pronounced effect on refining the pore structure of cement pastes at lower w/b ratios in comparison to higher w/b ratios [14]. Also discovered that the incorporation of metakaolin affected the porosity values of pervious mixes, irrespective of aggregate size. With an increase in metakaolin content, porosity values increased linearly, attributed to its filling effect.

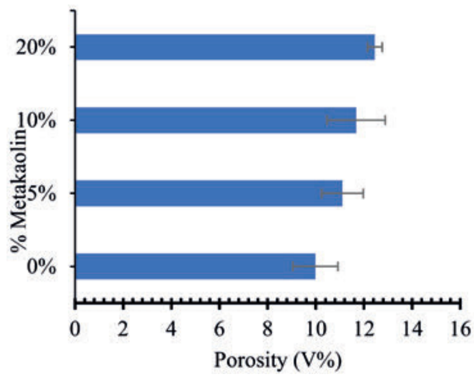

Fig. 1 Apparent porosity of the concrete mixtures
1. ábra Beton keverékek látszólagos porozitása

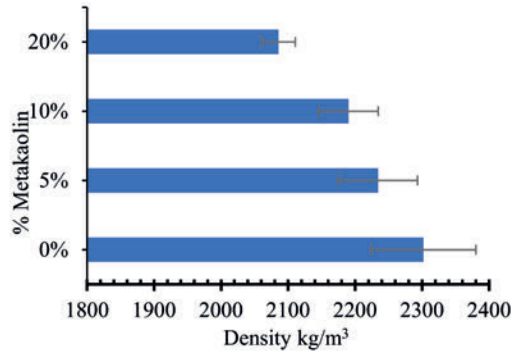

Fig. 2 Metakaolin effect on density of concrete
2. ábra Metakaolin hatása a beton sűrűségére

The density (ρ) of the specimens was determined per ASTM C1688-2014 after 28 days by dividing the dry weight by the volume. Samples were dried for four days, weighed, submerged in water, and their submerged weight recorded. The volume was calculated from the buoyant weight using the density of water. *Fig. 2* shows a significant density decrease with increased metakaolin content, except at 5% dosage, where density was higher than at 10% and 20%. This may be due to metakaolin reacting with calcium hydroxide during cement hydration, forming additional binding material (C-S-H gel). Thus, metakaolin substitution can help produce lightweight concrete [14]. Indicates that the addition of metakaolin had little effect on the concrete density, primarily because of its comparatively small volume in comparison to other ingredients [15]. It has been noted that when the replacement level reaches 20%, the rate of increase in density significantly decreases due to the prevalence of aggregate interlocking.

3.2 Compressive strength

Compressive strength (fc) was measured as the ratio of the failure load supported by the cube specimen to its contact surface area at a constant rate of 0.6 MPa/s. The results, averaged from 4 cube values, were tested at 14, 28, 48, 83, and 90 days. *Fig. 3* shows a significant increase in compressive strength with metakaolin substitution compared to the control mix. At 14, 28, 48, and 90 days, the 5% and 20% metakaolin mixes showed the highest compressive strength, although all replacement percentages performed better than the control mix [13]. Also observed that the pozzolanic reaction exhibited by metakaolin becomes more pronounced with age when substituted at a 5% level, surpassing the reactions seen at 10% and 20% replacement levels. On the other hand, [16] found that the interaction between metakaolin and $Ca(OH)_2$ leads to the formation of additional strength through the production of calcium silicate hydrates (C-S-H). When comparing the compressive strengths (fc) of the cube and half prism specimens, *Fig. 4* displays the interaction plot between the two.

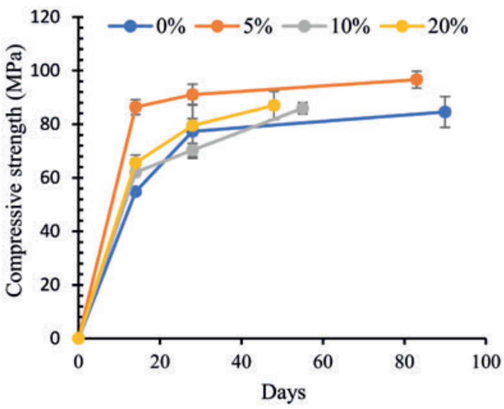

Fig. 3 Compressive strength of concrete cubes
3. ábra Beton kockák nyomószilárdsága

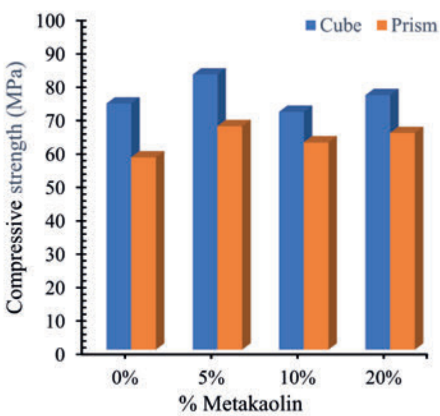

Fig. 4 Compressive strengths of cube and half prism
4. ábra Kocka és félhasáb nyomószilárdsága

From this it can be seen that overall, there isn't a significant difference in the compressive strengths of the cube and half prism specimens; however, on an individual basis, there is a noticeable difference in the compressive strengths of the cube and half prism specimens containing 0% and 5% metakaolin, while the specimens at 10% and 20% have relatively close compressive strengths. This relative rise in compressive strengths could be explained by giving the pozzolanic reaction adequate time to finish. For post-fire residual compressive strength, half prisms were tested after heating to 20 °C to 800 °C in a muffle furnace, following 28 days of natural drying and 4 days at 60 °C to remove moisture. *Fig. 5* and *6* show an initial rise in compressive strength between 200 °C and 300 °C, likely from the hydration of previously unhydrated metakaolin, followed by a rapid decrease after 400 °C, yet still comparable to the control mix. The 5% metakaolin mix maintained good post-fire strength, while the 20% mix showed a significant strength increase at 600 °C. No visible cracks were observed up to 600 °C, with microcracks appearing at 800 °C due to pressure from evaporating water. No spalling occurred, likely due to pre-drying. Maintaining compressive strength at high temperatures is crucial for fire-resistant concrete.

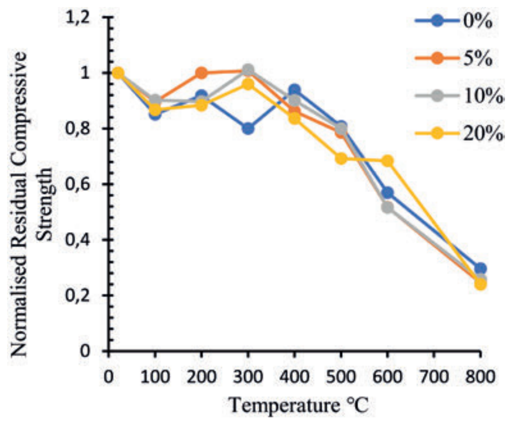

Fig. 5 Effect of temperature on compressive strength
5. ábra Hőmérséklet hatása a nyomószilárdságra

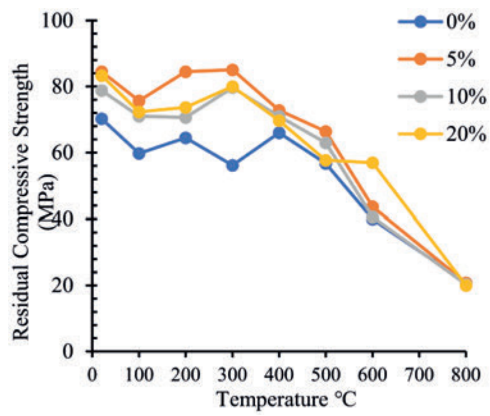

Fig. 6 Effect of temperature on compressive strength
6. ábra Hőmérséklet hatása a nyomószilárdságra

3.3 Flexure tensile strength

Flexural tensile strength test (*fl*) results at 40 days, depicted in *Fig. 7*, shows that higher proportions of metakaolin enhance the flexural tensile strength of the mixes. Notably, 5% and 20% metakaolin mixes demonstrated the most significant improvements compared to the control mix, while the 10% mix showed a decrease, but still better than the control mix, this is attributed to transition where the balance between cement hydration and metakaolin's pozzolanic reaction is less

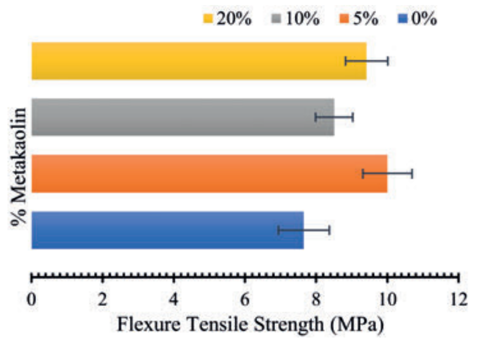

Fig. 7 Metakaolin effect on flexure tensile strength
7. ábra Metakaolin hatása a hajlító húzószilárdságra

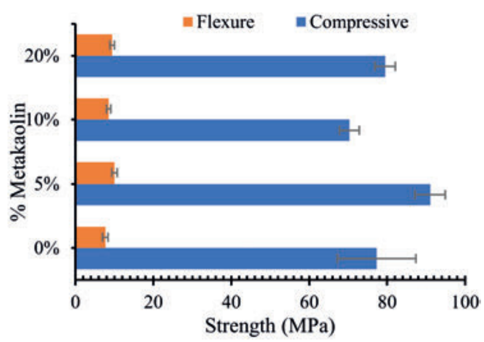

Fig. 8 Comparison of compressive and flexure strengths
8. ábra Nyomószilárdság és hajlító húzószilárdság összehasonlítása

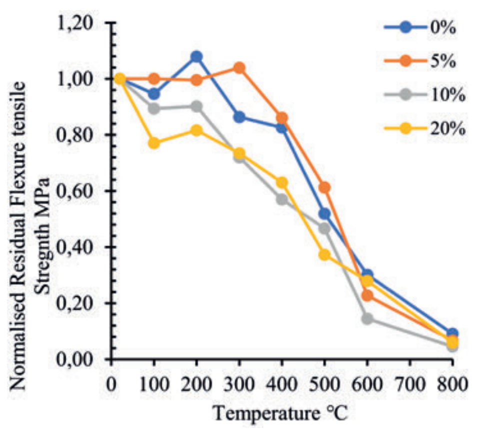

Fig. 9 Effect of temperature on flexure tensile strength
9. ábra Hőmérséklet hatása a hajlító húzószilárdságra

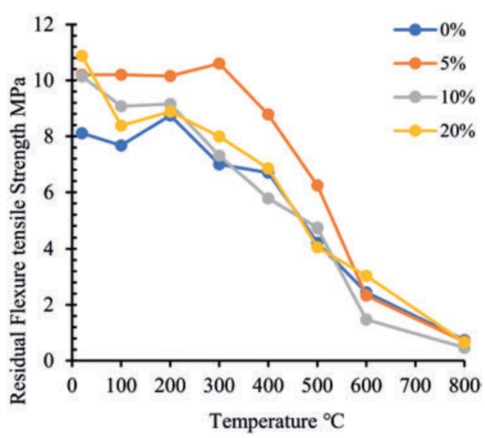

Fig. 10 Effect of temperature on flexure tensile strength
10. ábra Hőmérséklet hatása a hajlító húzószilárdságra

effective. This intermediate content may result in insufficient cement for full hydration while not providing enough metakaolin to enhance pozzolanic activity, leading to a weaker microstructure and poorer bonding between the cement paste and aggregates. *Fig. 8* illustrates that mixes with 5% and 20% metakaolin exhibit significantly higher strengths in both compressive and flexural tensile tests than the control mix, while the 10% metakaolin mix displays a slight decrease in flexural tensile strength and notable reduction in compressive strength compared to the control mix. After 28 days of drying, the impact of metakaolin on concrete's flexural tensile strength at elevated temperatures was assessed. Specimens were heated in an electric muffle furnace from 20 °C to 800 °C. Before the fire test, specimens were dried for approximately 4 days at 60 °C to remove accumulated moisture. *Fig. 9* and *10* show the flexural tensile strength of various mixtures at elevated temperatures, followed by the CMOD test. The flexural tensile strength of the 5% metakaolin mix was comparable to or higher than the control mix and significantly greater than the other mixes, exhibiting a notable increase at 300 °C. However, as the temperature increased further, the flexural strength decreased rapidly, with a slight decrease at 600 °C [17]. Noticed that the sharp increase in flexural tensile strength at 300 °C is primarily attributed to the substantial dehydration of the cement paste.

3.4 Fracture Energy

The fracture energy (*Gf*) was assessed using 72 prisms measuring 70 x 70 x 270 mm each, with a primary crack illustrated in *Fig. 11* [19]. This evaluation encompassed 48 prisms specimens of concrete containing metakaolin in the dosage of 10 and 20% and 24 prisms specimens of concrete without any additive. The fracture energy was determined by analyzing the crack mouth open displacement CMOD-load relationship plots obtained for each test specimen. This energy value was computed using the following equation.

$$Gf = \frac{0.75\,W0 + W1}{A\,lig} \tag{3}$$

$$W1 = 0.75\left(\frac{S}{L}\,m1 + 2m2\right) g.CMODc \tag{4}$$

Where GF - Fracture Energy, W0 - area under CMOD curve until rupture, W1- work done by dead load of jig, Alig – area of the broken ligament, S, L, m1, m2 according [19].

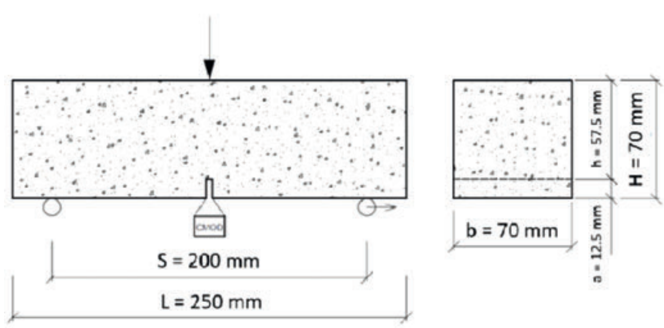

Fig. 11 (CMOD) arrangement [20]
11. ábra (CMOD) kísérleti elrendezés [20]

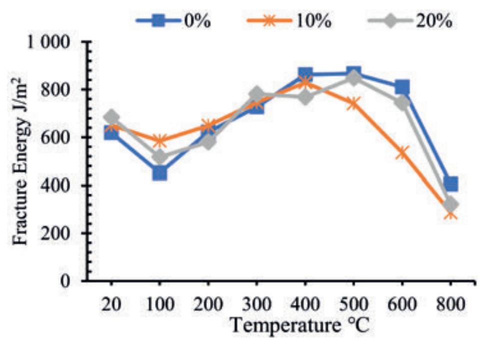

Fig. 12 Temperature effect on fracture energy
12. ábra Hőmérséklet hatása a törési energiára

Fig. 11 shows the modified experimental setup resembling three-point bending tests for flexural tensile strength, featuring a straight notch ending in a U-slot precisely cut at mid-span under point load. Notched three-point bending tests were conducted on each specimen until total failure, recording load-displacement curves at a rate of 0.01 mm/sec under CMOD control. Fracture energy (GF) was estimated by dividing the area under the load-displacement curves. *Fig. 12* displays average CMOD values at load bearing capacity of different mixes, showing a steady increase until reaching peak CMOD, where a slight drop occurred. Notably, a linear decrease in fracture energy was observed for the 10% mix starting from 500 °C, associated with thermal shock from rapid temperature rise. Microcracks in concrete began spreading at 300 °C, while breakdown of calcium hydroxide into calcium oxide and water occurred between 400 °C and 600 °C, peaking at 500 °C. However, over 600 °C, additional deterioration of the C-S-H gel is thought to be the cause of the deterioration of concrete's mechanical and physical characteristics [21]. Since metakaolin's particles are finer than cement's, adding it to the concrete mix can alter the packing density. Increased porosity in the mix leads to the formation of voids and microcracks, which can impact the concentration of stress areas and expedite the initiation and propagation of cracks. These defects lessen the concrete's fracture toughness and weaken it. [21, 22] Observed, the decline in (GF) observed in concrete exposed to heat is attributed to several factor. These include the development of microcracks within the concrete due to high temperatures, alterations in the pore structure, and changes in the existing pore network caused by the release of pore water and weakening of the ITZ (interfacial transition zone) bond due to the breakdown and evaporation of chemical water within the cement paste.

4. Conclusions

Based on the experimental study and observation the following conclusion has been drawn.

- Increasing metakaolin dosage generally increases porosity due to the rapid pozzolanic reaction with calcium hydroxide and changes in paste structure. Variations in curing conditions and water-cement ratio also influenced porosity.
- Density notably decreased with higher metakaolin content, but a slight increase was observed at 5% metakaolin compared to the control mix.
- Metakaolin substitution significantly improved compressive strength, with the greatest enhancement at 5% metakaolin due to rapid pozzolanic reactions and formation of calcium silicate hydrates (C-S-H).
- Flexural strength increased with higher metakaolin proportions; mixes with 5% and 20% showed significant improvements, while at the 10% replacement level, the mix reaches a transitional point where the balance between cement hydration and metakaolin reaction is suboptimal, leading to a reduction in flexure strength but still shows outstanding growth than the control mix.
- Compressive strength of specimens exposed to 20 °C-800 °C initially increased then decreased. Specimens with 5% metakaolin maintained notable post-fire compressive strength, similar to the control mix.
- No spalling was observed during the fire test at high temperatures, likely due to effective pre-test drying, which removed all water content and moisture.
- Concrete mixes with 5% metakaolin demonstrated significant flexural tensile strength compared to the control mix, with a significant increase at 300 °C due to substantial dehydration of the cement paste.
- Fracture energy initially increased linearly with temperature due to improved pore structure, but beyond 500°C, deterioration of the C-S-H gel led to decreased fracture energy.
- The study indicates that metakaolin substitution can enhance concrete's mechanical properties, including compressive and flexural strength, fracture energy, and resistance to water absorption and high temperatures. Mixes with 5% metakaolin showed the most favorable performance, highlighting its practical potential.

Acknowledgements

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