EFFECT OF USING METAKAOLIN ON FRESH AND HARD CONCRETE MIXES

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ABSTRACT

Cement industry account for 5-8% of worldwide CO_2 emission. To reduce the environmental impact of cement industries metakaolin and other pozzolanic materials are used to replace part of cement. In this study, metakaolin was obtained from local source. Slump test of the different concrete mixes conducted to explore the effect of incorporating metakaolin on fresh concrete properties. Compressive strength test, splitting tensile strength test and flexural strength test conducted to investigate the effect on hardened concrete properties, to find the optimum cement replacement ratio by metakaolin and to compare with silica fume. The experimental results showed that the use of metakaolin enhanced the workability of the fresh concrete, enhanced the hardened concrete mechanical properties compared to control mix and nearly the same as silica fume mix. The optimum replacement level was 15%, the enhancing in the concrete compressive strength was 10%, the reduction in the CO_2 emissions was 12.38%, and the energy saving was 5.56%, compared to that control mix.

Keywords: concrete, metakaolin, silica fume, compressive strength, tensile splitting strength, flexure strength

INTRODUCTION

Concrete is one of the most popular extensively used construction materials in the world. The concrete industry produces approximately 12 billion tones of concrete and uses about 1.6 billion tones of Portland cement worldwide each year (Malhotra and Metha 2005). At the same time, with the extensively use of Portland cement, the main component of the concrete, there are environmental concerns in both terms of intensive energy exhausted and global warming of carbon dioxide (CO_2) emission by cement manufacture, the manufacture of one ton of cement approximately 1.0 ton of CO_2 are launched into the atmosphere (Rashad and Zeedan, 2011). The cement industry account for 5-8% of worldwide CO_2 emission (Scrivener and Kirkpatrick, 2008). These cause serious environmental impact. In addition to consuming considerable amount of virgin materials (limestone and sand) and energy (energy demand about 4.69 GJ/ton). To reduce the environmental impact of cement industries metakaolin and other cementitious materials are used to replace part of cement or as a source of new cementless materials. Metakaolin reacts chemically with hydrating cement to form modified paste micro-structure. In addition to its positive environmental impact, metakaolin improves concrete workability, mechanical properties and durability. The term of metakaolin pozzolan refers to a silicious material which in-finely divided from and in the presence of water, reacts chemically with calcium hydroxide to form cementitious compounds.

Using the metakaolin in the concrete industry has received considerable attention in the recent years, using the metakaolin can achieve energy saving and reduction in the CO_2 emissions. However, although Egypt have a large reserve of kaolin , the metakaolin is not widely produced and used in the concrete industry due to the lack of adequate local experiments and studies about Egyptian metakaolin and comparing with the silica fume.

Accordingly, the main objectives of the current study are: investigate the effect of using the metakaolin on the fresh and hardened concrete mixes**,** identify the optimum replacement of metakaolin to the concrete mixes**,** compare the performance of the metakaolin with the silica fume, studying the environmental impacts of using the metakaolin considering the consumed energy and CO_2 release comparing to Portland cement**.**

MATERIALS AND METHODS

The experimental program was designed to achieve the research objectives of this study. The experimental program consists of two phases. Phase I, includes studying the physical and chemical characteristics of metakaolin and comparing their properties with the standard. Phase II, include studying the effect of using metakaolin as a partial replacement of cement on the properties of fresh and hardened concrete, in addition to one mix with 10% silica fume as cement replacement for comparison. The concrete mixtures were used in Phase II, incorporated metakaolin as a partial replacement of cement with replacement levels 10%, 15% and 20% (three mixes). Slump test of the different concrete mixes was carried out according to specification to explore the outcome of incorporating metakaolin on the properties of fresh concrete. Compression test, splitting tension test and flexure test were accomplished according to specifications respectively to investigate and compare the mechanical properties of the hardened concrete.

Properties of the used materials

Cement: The used cement in this study was Portland cement (CEM I 42.5 N) provided by BENI SUEF Company complying with ES 4756-1.

Fine Aggregate : The fine aggregate utilized in this study was natural siliceous sand with medium grading complying with ES 1109

Coarse aggregate: The coarse aggregate used was natural crushed stone, with particle size of 5-20 mm complying with ES 1109

Silica Fume: Silica fume was provided from Egyptian Ferro-Alloys Company (EFACO). Table 1 and 2 show the physical and the chemical composition of the used silica fume respectively, as obtained from the manufacturer.

Table(1): Physical properties of the silica fume (Provided by Manufacturer)

Property	Results
Specific surface area (m^2/g)	13.97
Specific gravity	2.20
Color	Dark gray

Table(2): Chemical composition of silica fume (wt %) (Provided by Manufacturer)

Oxide	SiO2	Al2O3	Fe2O3	CaO	MgO	K2O
SF	96.8	0.41	1.30	0.98	0.14	0.87

Metakaolin : Metakaolin was provided from the Aluminum Sulphate company of Egypt (ASCE).

Mixing Water: The water used in the mix design was potable water from the water supply network system.

Superplasticizer

Sikament R2004 was the used chemical addition to reduce the mixing water which confirming with ASTM C 494 type G and BS 5075 part 3 requirements. According to data sheet,

the superplasticizer was used to increase workability of concrete, long lasting control of slump loss, up to 20% water reduction. The properties of the used superplasticizer are revealed in Table 3.

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Table(3): The properties of the used superplasticizer "Provided by the manufacturer".

Item	Property
Color	Brown liquid
Chloride	Free
Density (at 20o C)	1.200 (kg/l)
Dosage	0.6-2.5% of weight of cement

Determination of physical and chemical properties of metakaolin

XRF Analysis of metakaolin: The chemical properties for metakaolin were analyzed by the X-ray fluorescence (XRF) analysis.

Particle Size Distribution of Metakaolin: The particle size distribution for metakaolin was analyzed by HORIBA, Laser scattering particle size distribution analyzer particle.

XRD Analysis of Metakaolin: In order to check the efficiency of the thermal treatment (Calcinations) of the raw kaolin (kaolin to be converted to metakaolin), X-ray diffraction (XRD) analysis was performed.

Mix Proportions: The control mix was designed to meet 30 MPa using natural course and fine aggregates. Five concrete mixes incorporating using metakaolin were used in this study. All mixes had a constant cement content of 350 kg/m3, constant W/C = 0.56, and constant content of superplasticizer (1.77% by cement weight).The concrete mixes consisted of three mixes incorporating metakaolin as partial substitute of cement with replacement levels of 10%, 15% and 20%. In addition to one mix incorporated silica fume as cement replacement with level 10% of cement replacement, and the control mix without metakaolin the mix proportions of the different concrete mixes are given in Tables 4. Concrete mixes were identified according to the metakaolin replacement level.

Table(4): Mix proportion of all mixes

Mix ID	Cement (Kg/m^3)	Silica fume (Kg/m^3)	MK (Kg/m^3)	Crushed Stone (Kg/m^3)	Sand (Kg/m^3)	Water (l/m^3)	Super-Plasticizer (l/m^3)
C	350	0	0	1128	752	198.7	6.2
R10	315	0	35	1123	749	198.7	6.2
R15	297.5	0	52.5	1121	747	198.7	6.2
R20	280	0	70	1119	746	198.7	6.2
S10R	315	35	0	1120	746.8	198.7	6.2

Mixing, Casting and Curing: A rotational mixer was utilized for concrete mixing. The fine aggregate was added to coarse aggregate inside the mixer before incorporating the cement. Half the amount of water content was added just a few seconds after the mixer had been turned on. One minute later the superplasticizer was added to the mixture during the addition of the remaining water and the whole ingredients were mixed for three minutes. Concrete was molded in the prepared molds in three layers and compacted using a steel bar, specimens were demolded after 24 hours and were wet cured till testing day. Three replicates were cast for each test and the average value will be used throughout the study.

Testing

Slump test: Slump values were recorded to determine the workability of fresh concrete for each mix according to ES 8411-2:2020.

Compression Strength Test: Three cubes of dimensions 150x150x150 mm from each concrete mix were examined at the age of 7, 28, 56 and 90 days using the 100 ton capacity compression testing machine. The test was carried out according to ES 1658-6:2018.

Splitting Tensile Strength Test: Three cylinders of 150 mm diameter and 300 mm height for each concrete mix were tested at the age of 28 and 90 days. The test was conducted according to ES 1658-9:2018.

Flexure Strength Test: Three beams of dimensions 100x100x500 mm for each concrete mix were tested in flexure at the age of 28 and 90 days using three points loading test. The test was examined according to ES 1658-5:2018.

RESULTS AND DISCUSSION

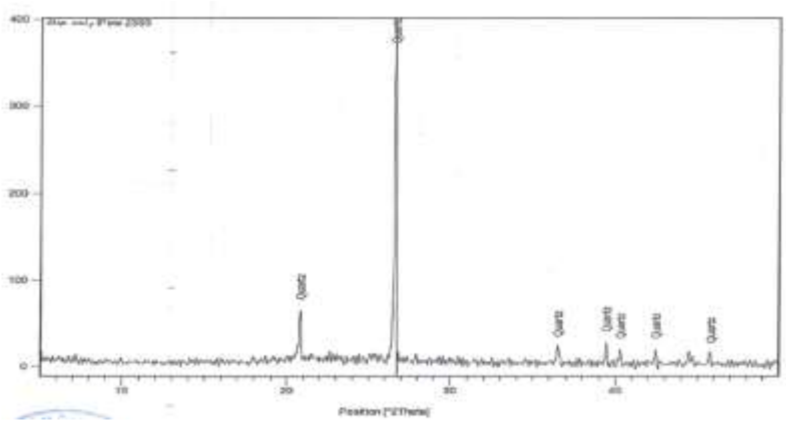
The Chemical composition (XRF) analysis of metakaolin: The main chemical composition of metakaolin were 52 % Silicon dioxide (SiO_2), 31 % aluminum oxide (Al_2O_3) and 2.0 iron oxide (Fe_2O_3) as shown in Table 5, which is complying with ES 8472-1:2021.

Table(5): Chemical composition of Metakaolin

Chemical component	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	TiO_2	P_2O_5
Value (%)	52	31	2.00	4.16	0.88	2.09	0.09	0.07	2.33	0.06

XRD analysis of metakaolin: The mineral composition using the X-ray diffraction (XRD) analysis was performed to determine the efficiency of the thermal treatment (Calcinations) of the raw kaolin (kaolin to be converted to metakaolin).

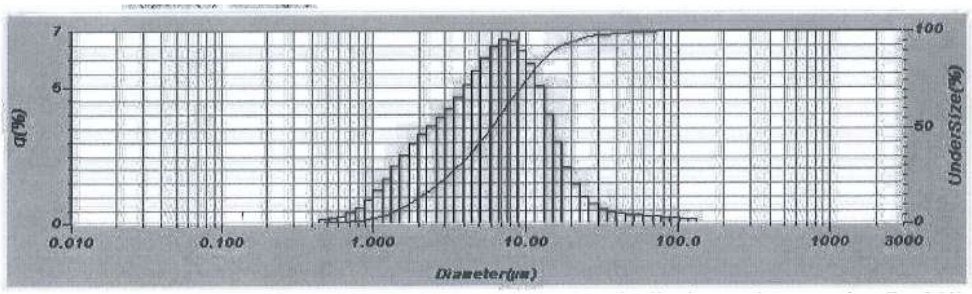
The XRD analysis results of the metakaolin shows that the compound name is quartz and the chemical formula SiO_2.



Figure(1): XRD analysis of Metakaolin

It was noticed that the only peak was for quartz and disappearance of peaks of kaolinite or any other clay mineral with amorphous background of metakaolin, which means that the thermal treatment (calcinations) was completed and all kaolin was converted to metakaolin.

Particle Size Distribution of metakaolin: The particle size distribution of the metakaolin is shown in Figure 2. The mean particle diameter is 8.9 µm. the metakaolin particle size fulfill the requirements of ES 8472-1:2021



Figure(2): Particle size distribution of Metakaolin

Test Results of Phase II: Phase II studies the properties of fresh and hardened concrete incorporating metakaolin as partial cement replacement

Test results of Fresh Concrete (Slump Test): Concrete workability was determined by using the slump test, which is the most important test for investigating the workability of the fresh concrete. Slump test results of the different concrete mixes are listed in Table 6.

Table(6): Slump test results of concrete mixes with metakaolin as cement replacement

Mix ID	Slump (cm)
Control	19
10%MK-R	21
15%MK-R	21.5
20%MK-R	22
10%S-R	19

From the above results of Table 6, it was found that the slump increased with the increasing of the metakaolin replacement percentage by using the same w/c for all mixes (0.56) and superplasticizer dose 1.77%.

In agreement with our results, Qian and Li (2001) showed the relationship between the metakaolin content and the slump value in fresh concrete. For concrete with a 1% superplasticizer addition, the slump progressively decreases with increasing metakaolin content. However, by increasing the superplasticizer dose to 1.2%, the slump showed only minor variation with increasing metakaolin content. On the contrary, Khatib (2008) and Johari et al. (2011) found that the presence of MK reduces the workability.

Test results of hardened concrete: The properties of the hardened concrete in this study were determined by using three tests, compressive strength test, splitting tensile strength test and flexure strength test.

Compressive strength: The compression test results at 7 days, 28 days, 56 days and 90 days of different concrete mixes are listed in Table 7.

Table(7): Average compressive strength

Mix.	Average Compressive Strength (N/mm2)			
	7 days	28 days	56 days	90 days
Control	29.11	33.87	38.6	42.1
10% MK-R	26.5	34.70	38.6	43.6
15% MK-R	25.1	38.9	42.5	46.7
20% MK-R	23.0	34.5	39.6	44.10
10% Silica-R	26.81	41.56	45.64	49.0

It was observed decreasing the average compressive strength of the 10% MK-R mix by 9% compared to the control mix at 7 days which may due to not starting the pozzolanic reaction of the metakaolin, and the 10% MK-R mix at age 28 days, 56 days and 90 days have no significant change compared to the control mix (<5%). The 10% MK-R mix at age 7 days almost the same as 10%Silica-R, the 10% MK-R mix decreased by 17%, 15% and 11% compared to the 10%Silica-R at ages 28 days, 56 days and 90 days respectively which may due to the higher activity of the silica fume compared to the metakaolin.

The 15% MK-R mix decreased by 14% compared to the control mix at 7 days and increasing the average compressive strength of the 15% MK-R mix at age 28 days, 56 days and 90 days compared to the control mix with percentage 16%, 10% and 11% respectively, the 15% MK-R mix at age 7 days decreased by 6% compared to 10% Silica-R may due to the higher cement content in the 10% Silica-R compared to the 15% MK-R, the 15% MK-R mix decreased by 6%, 7% and 5% compared to the 10% Silica-R at ages 28 days, 56 days and 90 days respectively, which may acceptable results considering that the price of the metakaolin is much lower than silica fume price.

The 20% MK-R mix decreased by 21% compared to the control mix at 7 days, the 20% MK-R mix at age 28 days, 56 days and 90 days have no significant change compared to the control mix (<5%), the 20% MK-R mix at age 7 days decreased by 14% compared to 10%Silica-R may due to the higher cement content in the 10%Silica-R compared to the 20% MK-R,

The 20% MK-R mix decreased by 17%, 13% and 10% compared to the 10%Silica-R at ages 28 days, 56 days and 90 days respectively, which means that 15% MK-R achieved better results than 20% MK-R. It was observed that the 15%MK-R is the optimum replacement mix achieving the maximum compressive strength at ages 28 days, 56 days and 90 days compared to 10%MK-R and 20%MK-R.

Compared to the results of previous studies, Wild *et al.*, (1996) found that 20% is the optimum replacement level of metakaolin and achieved improvement in the strength with 17.5% at age 90 days. They showed a maximum effect of the pozzolanic reaction between 7 and 14 days. Interestingly, Qian and Li (2001) found that 15% is the optimum replacement level of metakaolin and achieved 34% improvement in the strength at age 60 days. On the other hand, Courard *et al*., (2003) found that the optimum replacement level of metakaolin between 10% and 15% and achieved improvement in the strength with 23% at age 28 days.

In agreement with our results, Lee *et al*., (2005) found that 15% is the optimum replacement level of metakaolin improving the strength by 5% and 10% at ages 28 days and 90 days respectively. However, Badogiannis *et al*., (2005) found that 10% is the optimum replacement level of metakaolin comparing to 20% replacement level and the pozzolanic reaction of metakaolin is accelerated between 7 and 28 days.

Poon *et al*., 2006 found that 10% is the optimum replacement level of metakaolin comparing to 20% replacement level and improved the strength by 17.4% and 18.5% for w/c 0.3 and 0.5 respectively at age 90 days, knowing that the silica fume improved the strength by 12.8% and 14.4% for w/c 0.3 and 0.5 respectively at age 90 days thus disagreeing that the metakaolin improvement is higher than silica fume improvement. However, they found that he performance of the MK used in that study was superior to the SF in term of the strength development of concrete.

In the same line with our results, Khatib (2008) found that 15% is the optimum replacement level of metakaolin except that improvement in the strength with 27.6% occured at age 56 days. the maximum contribution of MK to strength occurs at 14 days of curing.

In opposition to our results, Vejmelkova *et al.*, (2010) found that 10% is the optimum replacement level of the metakaolin. However, in agreement with our results, the measurements of a wide set of properties of high-performance concrete containing 10% of Czech metakaolin as substitute of Portland cement confirmed that metakaolin could effectively serve as an alternative to silica fume and granulated blast furnace slag in the countries where these are either not available or economically in convenient.

Said-Mansour *et al*., (2011) found that found that 10% is the optimum replacement level of metakaolin comparing to 20% replacement level and improved the strength by 10%. Parallely, Megat Johari *et al*., (2011) found that 10% is the optimum replacement level of metakaolin and achieved improvement 11.6% in the strength at age 90 days, knowing that silica fume achieved

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30% strength improvement at the same age (90 days). The maximum contribution to strength takes place between the ages of 14 and 28 days at replacement level of 10% and 15%. On the other hand, Rashwan *et al*., (2015) found that the optimum replacement level of metakaolin between 15% to 20% and improved the strength by 25% for w/c 0.4.

Splitting Tensile Strength: The splitting tensile strength test results at 28 days and 90 days of different concrete mixes are listed in Table 8.

Table(8): Splitting tensile strength

Mix no.	**Splitting Tension Strength (N/mm2)**	
	28 days	**90 days**
Control	2.592	2.83
10%MK-R	2.598	2.81
15%MK-R	2.817	3.0
20%MK-R	2.779	2.97
10%Silica-R	2.752	3.04

It was found that the 10% MK-R mix at 28 days and 90 days have no significant change compared the control concrete mix. The 10% MK-R mix decreased by 6% and 8% compared to the 10%Silica-R at ages 28 and 90 days respectively. The 15% MK-R mix increased by 9% and 6% compared to the control mix at ages 28 days and 90 days respectively. The 15% MK-R mix at 28 and 90 days have no significant change compared the 10%Silica fume cement replacement mix (<5%).

The 20% MK-R mix increased by 7% and 5% compared to the control concrete mix for 28 days and 90 days respectively. The 20% MK-R mix at 28 days and 90 days have no significant change compared the 10%Silica fume cement replacement mix (<5%).

The 15%MK-R is the optimum replacement mix achieving the maximum tensile splitting strength at ages 28 days and 90 days compared to 10%MK-R and 20%MK-R,

Oppositely, Qian and Li (2001) found that the splitting tensile strength improved by 16% and 28% for replacement levels 10% and 15% respectively at age 28 days. However, in agreement with our results, AlMattar *et al*., (2009) found that the splitting tensile strength improved by 1% and 10% for replacement levels 10% and 15% respectively at age 28 days for w/c 0.55, while at w/c 0.4, the improvement was 10% for both metakaolin and silica fume 10% replacement level.

Flexural strength: The results of the flexural strength values at 28 days and 90 days of different concrete mixes are listed in Table 9.

Table(9): Flexural strength

Mix ID.	**Flexural Strength (N/mm2)**	
	28 days	**90 days**
Control	6.53	6.98
10%MK-R	6.72	7.17
15%MK-R	6.87	7.25
20%MK-R	6.66	7.12
10%Silica-R	7.11	7.53

It was found that the 10% MK-R mix at 28 days and 90 days have no significant change (<5%) compared the control concrete mix. The 10% MK-R mix decreased by 5% compared to the 10%silica-R at ages 28 and 90 days. The 15% MK-R mix increased by 5% compared to the control mix at ages 28 days, and have no significant change at age 90 days (<5%).

The 15% MK-R mix at 28 and 90 days have no significant change compared the 10%Silica fume cement replacement mix (<5%). The 20% MK-R mix have no significant compared to the

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control concrete mix (2%). The 20% MK-R mix decreased with 6% and 5% compared the 10%Silica fume cement replacement mix for the age 28 and 90 days respectively. The 15% MK-R is the optimum replacement mix compared to 10% MK-R and 20% MK-R,

Oppositely, Qian and Li, (2001) found that the flexural strength improved by 30% and 37% (disagree) for replacement levels 10% and 15% respectively at age 28 days and improved by 13% and 24% for replacement levels 10% and 15% at ages 80 days. However, in agreement with our results, Courard *et al*., (2003) found that the flexural strength improved by 3.8%, 3.6% and 0% for replacement levels 10%, 15% and 20% respectively at age 28.

Environmental Consideration of Using Metakaolin: Environmentally, Portland cement is not very friendly material due to CO_2 emissions into the atmosphere and energy consumption. Engineers must reduce its use in concrete and other alternatives must be used to replace cement. Using metakaolin as cement replacement material achieves environmental benefits. The CO_2 release from MK production comes only from the process of extraction of raw materials, and calcination at low temperature for de-hydroxylation).

Table 10 presents the values concerning energy consumption and CO_2 release into the atmosphere, without factoring in the transport of raw materials. Energy and CO_2 release from metakaolin production is calculated for flash calcinations (F. Cassagnabe're *et al*., 2010). Regardless of the concrete application, replacing cement with metakaolin gives similar performances on mechanical criteria, and has a positive effect on the environment because replacing cement with metakaolin offers significant energy savings in terms of raw materials production (1.86% to 7.42%) and reduced CO_2 release (4.125% to 16.5%) for (5% to 20%) cement replacement.

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Table(10): Consumed Energy and CO_2 Release of Cement Production Compared with metakaolin

Item	Portland Cement	Metakaolin
CO_2 release (kg/1000kg)	1000	175
Consumed energy (GJ/1000kg)	4.69	2.95

Table(11): Reduction of CO_2 Emission for Metakaolin / Concrete Mixes

Mix	CO_2 release (kg/1000kg)	Benefit (%)
MK - 0% (Control)	1000	---
MK - 10%	917.5	8.25
MK - 15%	876.25	12.375
MK - 20%	835	16.5

Table(12): Energy Saving of using Metakaolin / Concrete Mixes

Mix	Energy Consumption (GJ/1000kg)	Saving (%)
MK - 0% (Control)	4.690	---
MK - 10%	4.516	3.71
MK - 15%	4.429	5.56
MK - 20%	4.342	7.42

CONCLUSIONS

The following conclusions can be derived from the current work:

- ❖ The slump increased with the increasing of metakaolin replacement percentage for the used w/c (0.56) and superplasticizer dose 1.77%.
- ❖ The optimum dosage of metakaolin as a partial cement replacement is 15% enhancing the concrete compressive strength with 10% compared to the control mix (except at age 7 days),

enhancing the splitting tensile strength by 6% compared to control mix and the flexural strength have no significant change compared to the control mix.

- The mix with 15% metakaolin as cement replacement (15%MK-R) decreased by 7% compared to the 10% silica fume cement replacement mix (10%Silica-R), the splitting tensile strength and the flexural strength have no significant difference compared to the 10%Silica-R mix,
- Using 15% cement replacement by metakaolin in the concrete has a positive effect on the environment, by reducing the CO2 emissions from 1000 to 876.25 kg per every ton (12.375%), and saving in the consumed energy from 4.69 to 4.429 GJ per every ton (5.56%).
- Egyptian metakaolin can be considered better economically than silica fume, where the price per ton of metakaolin 3000 LE at the latest, while the price of silica fume 22 LE per kg.

RECOMMENDATIONS

- The key in producing metakaolin for use as pozzolanic material is the thermal treatment "calcinations" process to be completed and without over heated, where the overheating leads to non reactive materials, the kaolin used in this study was calcined at 750 o C for 1.5 hours.
- The average particle size of the metakaolin should be less than 45 µm.

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تأثير إستخدام الميتاكاولين على الخلطات الخرسانية الطازجة والمتصلدة

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المستخلص

صناعة الاسمنت تنتج ٥–٨% من انبعاثات الكربون عالميا. ولكي نقلل الاثار البيئية لصناعة الاسمنت ، الميتاكاولين ومواد بوزلانية اخرى تستخدم فى الاحلال الجزئى للاسمنت. فى هذه الدراسة ، تم الحصول على الميتاكاولين من مصدر محلى. تم إجراء اختبار الهبوط للخلطات المختلفه للتحقق من تأثير استخدام الميتاكاولين على خواص الخرسانة الطازجة. تم إجراء اختبارات مقاومة الضغط ومقاومة شد الانفلاق ومقاومة الانحناء للتحقق من تأثير استخدام الميتاكاولين على خواص الخرسانة المتصلدة ومعرفة النسبه المثلى للاحلال بالميتاكاولين والمقارنة مع غبار السليكا.

تبين من خلال الدراسة ان الميتاكاولين ادى الى تحسن فى التشغيلية للخرسانة الطازجة وتحسن فى الخواص الميكانيكية للخرسانة المتصلدة بالمقارنة مع الخلطة المرجعية وتكاد تتساوى مع غبار السليكا وان النسبة المثلى للاحلال بمادة الميتاكاولين كانت ١٥%، حيث حسنت فى مقاومة الضغط بنسبة ١٠%، والتقليل فى انبعاثات الكربون كان بنسبة ١٢,٣٨%. والترشيد فى استهلاك الطاقة كان بنسبة ٥,٥٦% بالمقارنة بالخلطة المرجعية.

الكلمات الدالة: الخرسانة، الميتاكاولين، غبار السليكا، مقاومة الضغط، مقاومة شد الانفلاق، مقاومة الانحناء