EFFECT OF METAKAOLIN DEVELOPED FROM LOCAL NATURAL MATERIAL SOORH ON WORKABILITY, COMPRESSIVE STRENGTH, ULTRASONIC PULSE VELOCITY AND DRYING SHRINKAGE OF CONCRETE

Abdullah SAAND [a*], Manthar Ali KEERIO [b], Daddan khan BANGWAR [c]

[a] PhD, Prof.; Department of Civil Engineering, Quaid-e-Awam University of Engineering, Science & Technology, Nawabshah, Pakistan
E-mail address: *abdullah@quest.edu.pk*

[b] M.E., Assistant Prof.; Quaid-e-Awam University College of Engineering, Science & Technology, Larkana, Pakistan
E-mail address: *mantharali99@quest.edu.pk*

[c] M.E., Assistant Prof.; Quaid-e-Awam University College of Engineering, Science & Technology, Larkana, Pakistan
E-mail address: *skb_khan2000@yahoo.com*

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Abstract

The utilization of pozzolanic materials like metakaolin (MK) in cement mortar and concrete is growing in construction industry all around the world to reduce the CO_2 release into the atmosphere and reduce energy consumption. This study instigates the performance of concrete containing locally developed metakaolin in terms of workability, unit weight, compressive strength, ultrasonic pulse velocity and drying shrinkage of concrete. The Portland cement (PC) is replaced by inclusion of developed local metakaolin (calcined Soorh at 800°C for 2 hours duration) with dosages range; 5% to 25% with increment of 5% (by weight of cement). The investigation revealed that concrete made with 15% replacement of ordinary Portland cement (OPC) with locally developed metakaolin (MK) has significant influance on workability, compressive strength, ultrasonic pulse velocity measurements and drying shrinkage of concrete as compared to OPC concrete.

Streszczenie

Wykorzystanie materiałów pucolanowych takich jak metakaolin (MK) w zaprawie cementowej i w betonie jest coraz częstsze w przemyśle budowlanym na całym świecie w celu ograniczenia emisji dwutlenku węgla do atmosfery oraz redukcji zużycia energii. W artykule opisano właściwości betonu zawierającego lokalnie dostępny metakaolin w kontekście urabialności, ciężaru właściwego, wytrzymałości na ściskanie, szybkości rozchodzenia się fali ultradźwiękowej i skurczu od wysychania betonu. Cement portlandzki (PC) został zastąpiony mieszanką lokalnie dostępnego metakaolinu (Soorh odwodniony w temperaturze 800°C w czasie 2 godzin) ze zróżnicowanym dozowaniem; 5% do 25% ze stopniowaniem co 5% (w stosunku do ciężaru cementu). W badaniach udowodniono, że zastąpienie 15% tradycyjnego cementu Portlandzkiego (OPC) lokalnie dostępnym metakaolinem (MK) znacząco wpływa na urabialność mieszanki, wytrzymałość na ściskanie, szybkość rozchodzenia się fali ultradźwiękowej i na wielkość skurczu od wysychania, w odniesieniu do betonu wykonanego z cementu portlandzkiego (OPC).

Keywords: **Compressive Strength; Local Metakaolin; Mechanical Properties; Soorh; UPV.**

1. INTRODUCTION

The most widely used construction material in the world is cement due to abundant availability of raw material for production of cement and its comparatively low cost and concrete usefulness. [1]. World cement usage is estimated to grow from 2.2% in 2015, 3.7% in 2016, and continue near 4 percent growth through 2017–2018. World cement usage grew 4.6% in 2014 from 4.0 billion metric tons in 2013 to 4.3 billion metric tons [2]. In fact, to produce one tone of cement about 0.8 tonnes of CO_2 is released in the atmosphere [3], which is approximately 5-8% of CO_2, release [4]. For the production of cement SO_3 and NOx are released in addition to CO_2, which is source of the greenhouse effect and acid rain [3, 5]. For production of cement, in addition to serious environmental effects a lot of energy is required (approximately 1700-1800 MJ/tonne clinker) [3]. Environmental problems due to manufacturing of cement can be reduced by replacement of part of the PC clinker using pozzolanic materials in mortars and concretes [1, 6]. The utilization of kaolinitic treated clays as an alternative pozzolanic material, obtained from an abundantly available natural material, has recently increased significance especially in places where there is poor accessibility of other industrial wastes or costly transportation of industrial by-products [7, 8]. Generally, the clay deposits include a combination of different clay mineral like kaolinite, palygorskite and illite and a great percentage of impurities of non clay materials, such as quartz, anatase, calcite and sulfides. The quantity of these impurities can be reduced by heat treatment [8]. The manufacture of metakaolin MK, kaolinitic clays are calcined at temperatures ranging between 550 and 900°C , which is a very reactive pozzolanic material [9]. Metakaolin reacts with $Ca(OH)_2$ in the presence of water at ambient temperature creating a cement compound like C–S–H and hydrates of alumina [10]. The manufacturing of Metakaolin saves the energy and reduce the amount CO_2 release [1, 6, 11]. The utilization of supplementary cementitious materials (SCM) to replace some percentage of part cement is an effective way to reduce CO_2 release from cement factories [12, 13]. During calcinations of kaolinitic clays water is released in the atmosphere instead of CO_2 and less energy (500 to 800°C) is required to produce reactive pozzolanic material named as metakaolin [1]. With the inclusion of local metakaolin of Iran in concrete, significant improvements in mechanical properties at different ages and up to 180 days are observed as compared to the control concrete. It was found that 12.5% and 10% replacement of cement with metakaolin at w/b ratio 0.40 and 0.35 are the best replacement [14]. The compressive strength of the OPC concrete and the concretes with the inclusion of MK at (w/b) of 0.3 and 0.5 are investigated by C.S. Poon *et al*. It was concluded that the concrete with 15% replacement of cement by the MK had more compressive strength as compared to OPC concrete at various tested ages, mainly at the age of three days. [15]. The usage of mineral admixtures as supplementary cementing material in concrete has a tendency to increase better sustainability in construction industry. The results showed that there was a significant improvement in compressive strength with the inclusion of 15% metakaolin in concrete [16]. The use of metakaolin in Korea remained mostly in fire proof walls, however, in recent times application of metakaolin is found as a supplementary cementing material. The results of strength tests showed that the optimal replacement of cement with metakaolin is between 10% and 15% [17]. The outcome on compressive strength of concrete with inclusion of metakaolin in concrete is investigated by J. M. Khatib and J. J. Hibbert. The OPC was partly replaced with 0-20% by metakaolin and (w/b) was kept same as 0.5 for all mixtures. The results revealed that with the replacement of cement by MK, there is a significant improvement in strength, particularly at the early periods of curing [18]. The effect on compressive strength of concrete with inclusion of metakaolin and calcined kaolins (CKs) were analyzed by Kasim Mermerdas *et al.* The results revealed that 15% replacement of cement with calcined kaolins (CKs) was found to be the most effective for improvement of compressive strength of concrete at the ages of 28 and 90 days of curing [19]. Ping Duan *et al.* reported that at 10% replacement of cement with Metakaolin, compressive strength of concrete increases gradually especially at 180 days [20]. The compressive strength, Resistivity and UPV for mortar and concrete is investigated by A.K. Parande *et al.* The 15% replacement of cement with MK gives the best result of compressive strength, Resistivity and UPV as compared to other substitution levels and OPC concrete. However, more than 15% replacement of cement with metakaolin reduces the compressive strength, resistivity and UPV [21]. H.S. Wong and H. Abdul Razak studied that the compressive strength improvement obtained by the 10% replacement of cement with metakaolin was 13.5% more than the ordinary concrete at the age of 90 days [22]. Maximum ultra pulse velocity obtained at about 12.5% replacement of cement with MK for air cured specimens while for water cured specimens maximum ultra pulse velocity obtained at relatively lower replacement of cement with MK content of between 7.5% and 12.5% MK [23]. The shrinkage was investigated by Kasim Mermerdas et al by using 5% and 15% replacement of cement with metakaolin. The results revealed that 15% replacement of cement with metakaolin gave the less shrinkage as compared to control concrete [31]. The substitution of cement by MK (up to 15%) reduces drying shrinkage as compared to concrete without MK [27, 32-33]. The replacement of cement with 5-15% MK shows better values of UPV whereas in 20% MK specimen displayed lesser UPV data compare to 15% MK specimen. [34]. At 28 days, the HCC mixes that contain barchip fibre with the incorporation of 10% MK, 1% CNS and 1% epoxy and cured in sea water have the highest value of UPV, it was 8.88% above the control mixes cured in sea water and 7.17% above the base mix cured in sea water [35].

To decrease the manufacturing of cement to acquire decrease in CO_2 release, reduction in energy consumption, decrease in overall construction cost an environmental friendly pozzolanic material is required. The aim of this research is to examine the performance evaluation of concrete containing local developed metakaolin in terms of workability, unit weight, compressive strength, ultrasonic pulse velocity and drying shrinkage. The metakaolin is developed from local available natural material Soorh, which is still not investigated and introduced as metakaolin/pozzolan material to replace the cement to be used in concrete. The Soorh is a type of clay abundantly available in billions of tonnes in the vicinity of Thatta district, Sindh, Pakistan.

Table 1.
Physical and chemical properties of cement, Soorh and developed Metakaolin [24]

Constituent	% age by weight OPC	% age by weight of Soorh	% age by weight of produced metakaolin	Sum of produced Metakaolin: $SiO_2+Al_2O_3+Fe_2O_3$
SiO_2	20.78	55.89	62.18%	89.9%
Al_2O_3	5.11	23.51	21.67 %	
CaO	60.89	-------	3.01%	
MgO	3	3.53	3.41%	
Fe_2O_3	3.17	8.15	6.01%	
K_2O	-----	5.89	1.85%	
Na_2O_3	-----	1.89	1.03%	
TiO_2	-----	1.14	1.03%	
In_2O_3	-----	-----	0.8%	
LOI (%)	1.71	7.4	0.5	
Blaine (cm^2/g)	3008	2101	2339	
Specific gravity	3.15	2.64	2.60	

Table 2.
Minerals composition of Soorh and developed Metakaolin [24]

Minerals	Soorh (%)	Developed metakaolin (%)
Quartz Si_2O_5	47.1	36.3
Illite $K(Al_4Si_2O_9(OH)_3)$	27.4	42.9
Stevensite $CaO_2Mg_{2.9}Si_4O_{10}(OH)_2.4H_2O$	12.1	16.5
Kaolinite $Al_2Si_2O_5(OH)_4$	11.9	--
Calcite magnesium $MgO.06CaO_{0.94}(CO_3)$	0.8	4.3
Hematite Fe_2O_3	0.7	--

2. EXPERIMENTAL PROGRAM

2.1. Materials

Ordinary Portland cement and raw material Soorh (local natural material) is used. In concrete specimen, clean natural hill sand retained on sieve ≠ 4 as fine aggregate and coarse aggregate retained on ¾ inch sieve was used. Gradation curve of fine aggregate and coarse aggregate is shown in Fig. 1 and Fig. 2 respectively. In all mixtures of concrete the gradation of fine aggregate and coarse aggregate was constant. Physical properties and chemical composition of Soorh clay and of metakaolin obtained by thermal treatment of Soorh are shown in Table 1 and the mineralogical composition of natural material Soorh and developed metakaolin is presented in Table 2.

The mineralogical composition of natural material Soorh and developed metakaolin is determined by X-Ray diffraction (XRD). It is obvious that after the thermal treatment of Soorh, the amount of (raw impurity) quartz has reduced from 47.1% to 36.3% as shown in Table 2 [24]. Alejandra Tironi *et al.* studied that the compressive strength of mortar has improved by the 30% replacement of cement with thermally treated clays containing more than 40% quartz (raw impurity) [30].

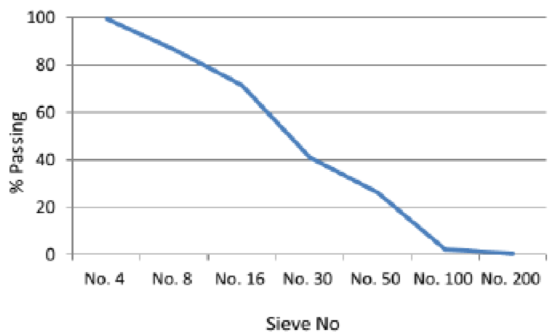

Figure 1.
Graduation curve of fine aggregate

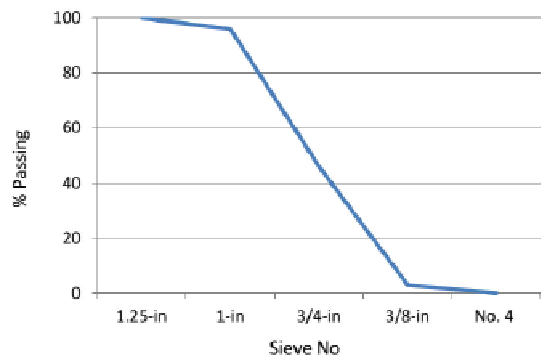

Figure 2.
Gradation curve of coarse aggregate

2.2. Mix proportions

OPC concrete and metakaolin modified concrete mixtures with w/b ratio of 0.55 and slump ranges of 25-50 mm were designed. For metakaolin concrete, the cement was partially replaced with 5%, 10%, 15%, 20% and 25% with developed metakaolin (calcined natural material Soorh at 800°C for 2 hours) by weight of cement. Accordingly, one mixtures of OPC concrete and five mixtures of metakaolin modified concrete were prepared for this study. Details of mix proportion of concrete are given in Table 3. The grading of the aggregate for all concrete mixtures was kept constant. All the mixtures of concrete were mixed as per ASTM C192 in revolving pan mixer. For every mixture 10 cylindrical specimen of 100 mm dia. 200 mm height for compressive strength test and 5 cubical specimens of 100 mm x 100 mm x 100 mm for Ultrasonic pulse velocity test were cast. For every concrete mix 5 concrete prisms of size 70 x 70 x 280 mm were cast to examine the free shrinkage and weight loss of control and metakaolin concrete.

2.3. Test Methods

Slump test as per ASTM C143 and unit weight, as per ASTM 138 were carried out. For 7 and 28 day compressive strength of the OPC concrete and metakaolin modified concrete, the compression test was carried out on the cylindrical specimens using universal testing machine as per ASTM C39 on 7 and 28 days curing specimen. The ultrasonic Pulse velocity test was carried out on the cubical specimens according to ASTM C597 – 09 at 28 days age. The Free shrinkage test was carried out as per ASTM C157 about 42 days. Free shrinkage test specimens were cured for 24 h at 20°C and 100% relative humidity before demoulding. After that, the specimens were exposed to drying in a humidity cabinet at 23±2°C and 50±5% relative humidity. The five specimens were used for each testing age and average value was recorded.

Table 3.
Mix Proportion of concrete

Concrete Mix	Cement Kg/m^3	MK Kg/m^3	Total Binder Kg/m^3	W/B	Water Kg/m^3	F.A Kg/m^3	C.A Kg/m^3	Slump (mm)
CM	346	--	346	0.55	190	692	1038	25-50
MK5	328.7	17.3	346	0.55	190	692	1038	25-50
MK10	311.4	34.6	346	0.55	190	692	1038	25-50
MK15	294.1	51.9	346	0.55	190	692	1038	25-50
MK20	276.8	69.2	346	0.55	190	692	1038	25-50
MK25	259.5	86.5	346	0.55	190	692	1038	25-50

3. RESULTS & DISCUSSION

3.1. Workability and unit weight of fresh concrete

The results of slump test of control and Metakaolin concrete are presented in Figure 3.

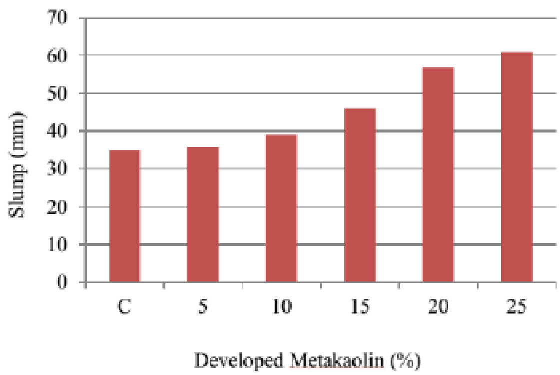

Figure 3.
Slump of control and metakaolin concrete

From the Figure 3, it is clear that the workability of metakaolin concrete is increased with the replacement of cement by calcined Soorh (developed metakaolin) as compared to control OPC concrete.

The increase in the workability of metakaolin concrete is due to lower specific surface area of local developed metakaolin than that of cement. As the specific surface area is less water demand shall be less and at same water cement ratio the workability shall be more.

The results of unit weight of control and Metakaolin concrete are highlighted in Figure 4.

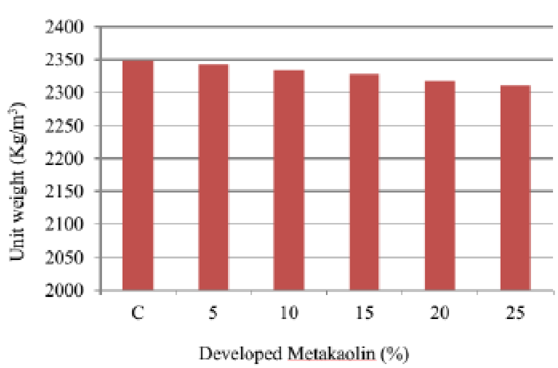

Figure 4.
Unit weight of plain and metakaolin concrete

It is obvious from the data presented in Figure 4, that the unit weight of Metakaolin concrete mixes is slightly decreased as compared to control mix with replacement of cement by calcined Soorh (developed metakaolin). The unit weight of metakaolin concrete is less due to less specific gravity of metakaolin than that of cement.

3.2. Compressive Strength

The comparison of 7 and 28 compressive strength of metakaolin concrete produced with replacement of cement (0-25%) with developed Metakaolin (calcined natural material Soorh calcined at 800°C for 2 hours duration) is highlighted in Figure 5.

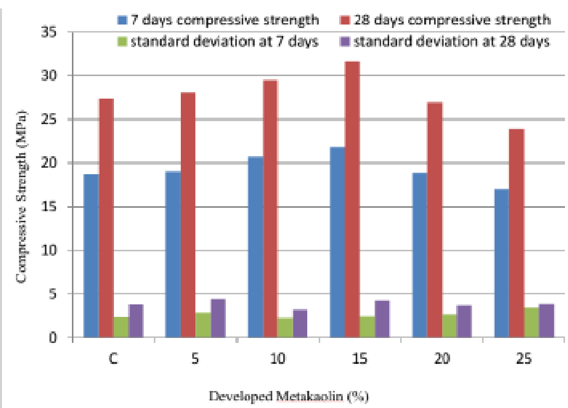

Figure 5.
Compressive strength of control and Metakaolin concrete at 7 and 28-Days

The Compressive Strength of Metakaolin concrete is increased than that of control mix with the replacement of cement by locally developed metakaolin; with the inclusion range 5% to 15 %. The maximum compressive strength, 31.65 MPa (i.e. 15.43% increase compared to control) at 28 days have been achieved at 15% substitution of cement by developed Metakaolin. On further substitution of cement with developed Metakaolin, the Compressive Strength of metakaolin concrete is decreased as compared to control concrete.

The most important aspects of MK to contribute in the strength are (i) the filling effect, (ii) the dilution effect, and (iii) the pozzolanic reaction of MK with CH [25]. Parande *et al.* also reported that the 15% substitution of cement by metakaolin gave maximum compressive strength as compared to ordinary concrete and replacement levels of cement with metakaolin [21]. The reduction in compressive strength for MK20 and MK25 as compared to MK15 is due to the effect of a clinker dilution effect [21]. The results revealed that 15% replacement of cement with calcined kaolins (CKs) was found to be the most effective replacement for improvement of compressive strength of concrete at the ages of 28 and 90 days of curing [19].

3.3. Ultrasonic Pulse Velocity of concrete

To investigate the internal integrity and quality of concrete with replacement of cement by local metakaolin ultrasonic pulse velocity test was carried out. The comparison of Ultra pulse velocity of concrete produced with replacement of cement (0-25%) with developed Metakaolin (calcined natural material Soorh calcined at 800°C for 2 hours duration) is presented in Figure 6.

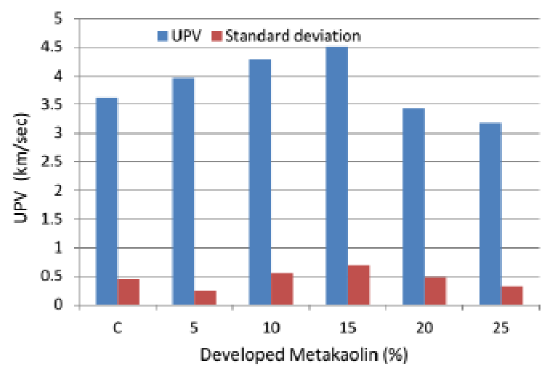

Figure 6.
Ultra pulse velocity control and Metakaolin concrete

The Ultrasonic pulse velocity of metakaolin modified mixes is increased as compared to CM with the replacement of cement by the local developed metakaolin, with 5% to 15 %. On further replacement of cement with more than 15% by developed Metakaolin, the UPV is reduced. A. K. Parnanade *et al.* also found that 5-15% replacement of cement with metakaolin has shown better resistivity and UPV than that of control concrete. While with 20% replacement of cement with metakaolin, it showed lesser resistivity and UPV measurements [21].

3.4. Drying, Shrinkage and Weight Loss

The comparison of drying and shrinkage of concrete produced with replacement of cement (0-25%) with developed Metakaolin (calcined natural material Soorh calcined at 800°C for 2 hours duration) is presented in Figure 7.

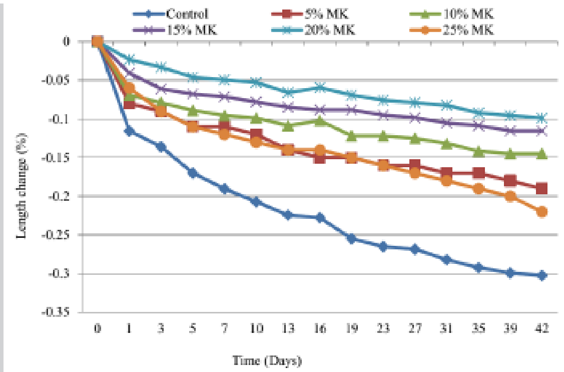

Figure 7.
Free shrinkage of control and metakaolin modified concrete

The shrinkage of metakaolin modified mixes is reduced as compared to CM with the replacement of cement by the local developed metakaolin, with 5% to 20%. On further replacement of cement with more than 20% by developed Metakaolin, the shrinkage is increased. In fact, the consequence of Metakaolin on shrinkage of cement pastes can be the effect of four phenomena: (i) cement dilution by Metakaolin, less cement creating less shrinkage, (ii) heterogeneous nucleation of hydrates on the surface of Metakaolin, faster cement hydration and, accordingly, increasing shrinkage, (iii) pozzolanic reaction of Metakaolin with CH produced by cement and, (iv) increase of capillary tension [27-29].

The drying shrinkage at 10% and 20% substitution of cement with the metakaolin were investigated by Guneyisi *et al.* The maximum reduction in shrinkage was observed at 20% replacement of cement with metakaolin. The remarkable reduction in shrinkage strain metakaolin or calcined kaolins modified concrete is due to the inclusion of thermally heated kaolins [26]. The effect with 5% and 15% replacement of cement with MK on drying shrinkage was studied by Gunysee *et al.* At 15% substitution of cement with metakaolin, it demonstrated significant reduction in shrinkage i.e. 42% lower shrinkage than that of plain concrete [16].

The comparison of weight loss of concrete produced with replacement of cement (0-25%) with developed Metakaolin (calcined natural material Soorh calcined at 800°C for 2 hours duration) is presented in Figure 8.

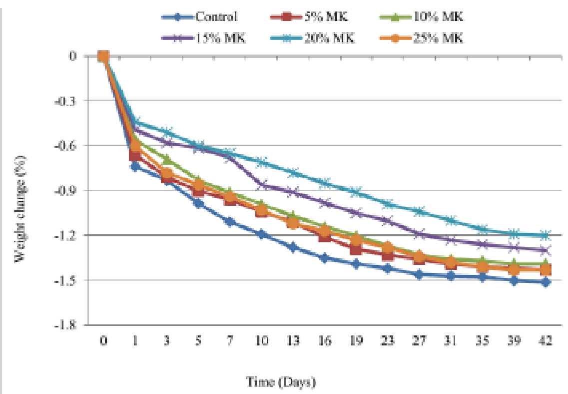

Figure 8.
Weight change of control and metakaolin modified concrete

Concrete mixes incorporated with mineral admixtures indicated lower weight loss similar to the drying shrinkage test results; inclusion of MK from 5% to 20% to the concrete mixes decreased the weight loss considerably. On further replacement of cement with more than 20% by developed Metakaolin, the weight loss is increased.

It was observed by Gunysee et al that after 42 day drying, depending mainly on replacement level, the MK exhibited up to 18% weight loss in comparison to plain concrete [16].

4. CONCLUSIONS

- The workability of all the metakaolin modified mixes is increased than that of control mix.
- The Unit weight of fresh concrete of all the metakaolin modified mixes is slightly decreased than that of control mix.
- The Compressive Strength of Metakaolin concrete is increased than that of control concrete with the replacement of cement by locally developed Metakaolin; ranging from 5% to 15% and on further substitution of cement the compressive strength is decreased.
- The Ultrasonic pulse velocity of modified mixes is increased as compared to CM with the replacement of cement by the locally developed metakaolin from 5% to 15% and on further substitution of cement by developed Metakaolin the UPV is reduced.
- The shrinkage and weight loss of metakaolin modified mixes is reduced as compared to CM with the replacement of cement by the local developed metakaolin, with 5% to 20%. On further replacement of cement with more than 20% by developed Metakaolin, the shrinkage is increased.
- On the bases of the results of workability, compressive strength, UPV and drying shrinkage, it can be concluded that the concrete with 15% replacement of cement by the locally developed metakaolin (calcined Soorh at 800°C for 2 hours duration) is optimum.

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