

Development of quaternary blended high performance concrete made with high reactivity metakaolin

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Abstract

In the last three decades, supplementary cementitious materials such as fly ash, silica fume and ground granulated blast furnace slag have been judiciously utilized as cement replacement materials as these can significantly enhance the strength and durability characteristics of concrete in comparison with ordinary Portland cement (OPC) alone. Hence, high-performance concretes can be produced at lower water/powder ratios by incorporating these supplementary materials. One of the main objectives of the present research work was to investigate synergistic action of binary, ternary and quaternary blended high strength grade (M80) concretes on its compressive strength. For blended high strength grade (M80) concrete mixes the optimum combinations are: Binary blend (95%OPC +5% FA, 95%OPC +5% MS and 95%OPC +5%MK), ternary blend (65%OPC+20%FA+15%MS) and quaternary blend (50%OPC+28%FA+11%MS+11%MK). Use of metakaolin in fly ash based blended concretes enhances compressive strength significantly and found to be cost effective in terms of less cement usage, increased usage of fly ash and also plays a major role in early strength development of fly ash based blended concrete.

Keywords: high performance Concrete, Metakaolin, Fly ash, quaternary blended concrete, high strength concrete..

1. Introduction

One of the effective methods to conserve the Mother Nature's resources and also reduce the environmental impact is to use Supplementary Cementitious Materials (SCMs) by substituting OPC partly or totally in concrete. Since most of SCMs are pozzolanic in nature and hence they are helpful in increasing later strength of concrete [1][2][4]. Blending of SCMs with cement has many advantages such as saving in cement, utilization of industrial by-products, enhancement of micro structural properties of concrete and reduces environmental impact through reduced greenhouse gases production. Most of the SCMs are industrial by-products which are considered as waste and pollutants when dumped into land or thrown into water bodies. So blending them in concrete becomes safe disposal method for them. Such SCMs are Fly ash (FA), Ground Granulated Blast furnace Slag (GGBS), Micro Silica (MS) or Silica Fume (SF), Copper slag (CS), Rice Husk Ash (RHA) etc.[3]

2. Objectives of the Present work

The primary objectives of this research work is to quantitatively comprehend and assess the role of optimum metakaolin (MK) in development of strength in binary, ternary and quaternary blended concrete, made with optimal micro silica and fly ash, of High strength grade (M80).

3. Experimental Investigations

The aim of the present experimental investigations is aimed to obtain specific experimental data which helps to understand the effect of synergic action of Metakaolin (MK), Microsilica (MS)

and fly ash (FA) combinations in blended concrete mixes of high strength grade (M80) on rheological behavior and strength. The experimental programme consisted of casting and testing specimens of high strength grade (M80) of binary, ternary and quaternary blended concretes made with Fly Ash (FA), Microsilica (MS) and Metakaolin (MK). Erntroy and shaklock empirical mix design was adopted to arrive at the suitable mix proportions and final quantities for the binary, ternary and quaternary blended concrete based on a number of trial mixes.

To accomplish the defined objectives, the scope of the work is framed into phases:

Phase 1: Physical and Chemical Properties of Materials Used

- Studies on physical and chemical properties of cement, coarse aggregate, fine aggregate (river sand), mixing water.
- Studies on physical and chemical properties of mineral admixtures such as fly ash, metakaolin, silica fume and that of chemical admixtures such as Superplasticizer.

Phase 2: Determination of Mix Proportions

Based on Erntroy and Shacklelock's empirical graphs, material quantities such as powder content (Cement + Pozzolan), fine aggregate, coarse aggregate, water and dosages of SP, required for 1 cu.m, are evaluated for High strength grade (M80) of concrete. Final quantities, for the above grade of concrete mixes considered, are assumed after several trial mixes on material quantities computed.

Phase 3: Optimization of pozzolans in concrete mixes

In the first part of this phase, based on the assumed final material quantities in Phase 2, the optimum proportions of fly ash (FA), micro silica (MS) and metakaolin (MK) combinations in binary, ternary and quaternary blended concrete mixes, that attain desired strength property, are identified through several trial mixes carried

out in the laboratory for the grade considered for study.[5][6][7][8][9]&[10].

Phase 4: Studies on Compressive Strength

Compressive strengths at 3, 7, 28 and 60 days were determined by conducting detailed laboratory investigations on high strength grade (M80) made with optimum quantities of FA, MS and MK combinations in binary, ternary and quaternary blended concrete mixes.

4. Materials Used

4.1 Cement

Ordinary Portland cement (OPC) of 53 grade [IS: 12269-1987, Specifications for 53 Grade Ordinary Portland cement] has been used in the study.

4.2 Fine Aggregates (River Sand)

The fine aggregate used was locally available river sand without any organic impurities and conforming to IS: 383 – 1970.

4.2 Coarse Aggregate

The coarse aggregate chosen for blended concrete was typically round in shape, crushed granite metal of size of 20 mm and 10 mm graded obtained from the locally available quarries was used in the present investigation.

4.4 Water

Water used for mixing and curing was potable water, which was free from any amounts of oils, acids, alkalis, sugar, salts and organic materials or other substances that may be deleterious to concrete or steel conforming to IS : 3025 – 1964 part22, part 23 and IS : 456 – 2000 [Code of practice for plain and reinforced concrete].

4.5 Fly Ash

Fly ash used in this investigation was procured from Vijayawada Thermal Power Station, Andhra Pradesh, India. It conforms with grade I of IS: 3812 – 1981 [Specifications for flyash for use as pozzolana and admixture].

4.6 Micro silica (MS)

Micro silica Grade 92D conforming to IS: 15388 -2003 is used. Silica fume has specific surface area of about 20,000m²/kg.

4.7 Metakaolin (MK)

Metakaolin obtained from KOAT manufacturing company, Vadodara, Gujarat has been used.

4.8 Super Plasticizer (SP)

For M80, BASF Glenium B233, High-performance super plasticizer based on PCE (polycarboxylic ether) for concrete conforming to IS: 9103-1999 is used as a water-reducing admixture.

5. Mix Proportioning

The mix proportioning was done based on the Erntroy and shaklock mix design approach for high strength grade (M80) of binary, ternary and quaternary blended concretes made with optimum combinations of fly ash (FA), microsilica (MS) and metakaolin(MK). Several trial mixes are conducted on number of blended concrete mixes made with the different possible combinations of Fly Ash (FA), Microsilica (MS) and Metakaolin (MK) to develop various binary, ternary and quaternary blended concrete

systems to determine the appropriate optimized quantities of Fly Ash.

6. Test Results and Discussions

The test results of experimental investigations carried out during the development of high strength grade (M80) binary, ternary and quaternary blended concrete mixes made with optimum proportions of fly ash (FA), microsilica (MS) and metakaolin (MK) combination are tabulated in the following sections. Quantities required for 1 cu.m are evaluated for high strength grade (M80) binary, ternary and quaternary blended concretes made with optimum proportions of Fly Ash (FA), Microsilica (MS) and Metakaolin (MK) combination based on calculations from Erntroy and shacklock mix design method. Final quantities, for all blended concrete mixes considered, are assumed after several trial mixes on quantities computed

Table 1. Quantities in kg per cu.m for high strength (M80) grade blended concrete obtained using Erntroy and shacklock Mix Design

	Cement	Fine Aggre- gate	Coarse Aggre- gate	Water
Quantity kg/m ³	700	644	966	150 L

The computed amount of OPC is 700 kg. But keeping in view the clause 8.2.4.2 of IS 456-2000, the maximum cement content is limited to 450 kg per cum of concrete. After trail mixes, revised quantities in kg per cu.m for high strength grade (M80) blended concrete mix are arrived without compromising the desired strength property.

The final quantities for high strength M80 grade blended concrete mix are tabulated in Table 2.

Table 2: Final Quantities for trial mixes of high strength M80 grade blended concrete mix

	Ce- ment	Total Pozzo- lana	Total Pow- der Con- tent	Fine Aggre- gate	Coarse Aggre- gate	Water (wa- ter/powder =0.23)
Quan- tity kg/m ³	450	250	700	644	966	150L

Henceforth, the total amount of powder quantity (cement + poz- zolanic mixture) adopted for high strength M80 concrete is 700 kg/m³ and water/powder ratio is 0.23 for all blended high strength M80 concrete mixes.

6.1 Optimization of SCMs proportions in blended concrete mixes

This phase identifies the optimum proportions of fly ash, micro silica and metakaolin in binary, ternary and quaternary blended concrete mixes in order to obtain the enhanced performance of blended concrete at all ages. The details of the quantities of materials, replacement percentages and quantities (kg) of SCMs and OPC in total powder content and their corresponding fresh properties are shown in Table 3 to Table 4 respectively for high strength grade (M80) of binary, ternary and quaternary blended concrete made with optimum proportions of Fly Ash (FA), Microsilica (MS) and Metakaolin (MK) combination.

Table 3 gives base quantities of high strength grade (M80) blended concrete mix derived after several trial mixes on the quantities calculated using Erntroy and shacklock mix design method. It can be observed that the total powder content is 700 kg/m³ with cement content restricted to 450 kg/m³ from durability of concrete point of view and rest of the powder is fly ash (250 kg/m³). Depending on the above calculated base quantities for high strength

grade (M80), twenty nine (29) blended concrete mixes were designed in three groups of binary, ternary and quaternary. Table 2 shows various blended high strength grade (M80) blended concrete mixtures made with different proportions of Fly Ash (FA), Microsilica (MS) and Metakaolin (MK). In Mix designation, number indicates percentage by weight of total powder content. Various binary, ternary and quaternary blended concrete mixes were prepared with different proportions of Fly Ash (FA), Microsilica (MS) and Metakaolin (MK). (B1 to B8, T1 to T8 and Q1 to Q12). Mix numbers B1 to B8 are binary blended concrete mixtures made of either fly ash (FA) or microsilica (MS) or metakaolin (MK) while Mix numbers T1 to T8 are ternary blended fly ash based concrete mixtures made of microsilica (MS) or metakaolin (MK) and Mix numbers Q1 to Q12 are quaternary blended fly ash based concrete mixtures made of microsilica (MS) and metakaolin (MK) combination.

In high strength grade (M80) concrete mix 'C1' developed with 100% OPC does not yield desired strength. So using 100% OPC in development of high strength grade (M80) concrete mix is completely ruled out. In binary blended high strength grade (M80) concrete mixtures, percentage replacement of fly ash by weight of total powder content is 35% i.e. 250 kg/m³ (B1) which is based on preliminary calculation from mix design. For the mix proportion C65+FA35, desired strength is not realized. For binary blended concrete mixtures made with percentage replacement of either micro silica (MS) or metakaolin (MK) or both combined, micro silica (MS) and metakaolin (MK) are limited to 5-15% and 5-20% respectively.

In ternary blended micro silica (MS) and fly ash (FA) blended high strength grade (M80) concrete mixtures (T1 to T4) percentage replacement of micro silica (MS) is limited to 5 -20% by weight of total powder content. Similarly in ternary blended metakaolin (MK) and fly ash (FA) blended high strength grade (M80) concrete mixtures (T5 to T8), percentage replacement of metakaolin (MK) is limited to 5 -20% by weight of total powder content. In both the above ternary blended MS+FA blended concrete and MK+FA blended concrete mixtures (T1 to T8), the cement content is kept constant (65% by weight of total powder content).

In binary blended high strength grade (M80), for fly ash (FA) blended concrete mix (B1) and metakaolin (MK) blended concrete mixes (B5 to B8) desired strength is not realized. But in binary blended micro silica (MS) blended concrete mix, desired strength is attained, if the MS percentage replacement is limited to 5-10% by weight of powder. The optimal mix chosen for binary blended micro silica (MS) based concrete mix is 5% MS replacement (B2). Henceforth, for high strength grade (M80) mixes, Mix OPC95+MS5 (B2) is taken as reference mix.

In ternary blended metakaolin (MK) and fly ash (FA) blended high strength grade (M80) concrete mixtures (T5 to T8), desired strengths are not obtained for any of the mixes. But for micro silica (MS) and fly ash (FA) blended ternary blended concrete mixes (T1 to T4), up to 15% MS by weight of powder, desired strengths are attained satisfactorily. So C65+FA20+MS15 (T3) concrete mix is considered optimal in ternary blended high strength grade (M80) concrete mixes.

In quaternary blended high strength grade (M80) concrete mixtures (Q1 to Q12) made of microsilica (MS) and metakaolin (MK) combination, keeping cement content constant (65% by weight of total powder content), microsilica (MS) and metakaolin (MK) proportions are limited to 7 – 14%.

For quaternary blended concrete mix (Q1), initially 7% MS and 7% MK replacements are assumed, keeping cement content constant i.e. 65% by weight of total powder content and rest of powder is fly ash, and required workability is not satisfied. So microsilica (MS) and metakaolin (MK) are gradually increased to 14% each yet workability is not achieved. Then author proposed to additionally increase fly ash content incrementally by 10% by weight of powder content (700 kg/m³), thereby incrementally increasing the powder quantity by 70 kg. With addition of 30% of fly ash (FA) to the C65+FA7+MS14+MK14 concrete mix (Q11), required workability and strength properties are achieved. So for

quaternary blended concrete mix, the optimum combination of cement and pozzolanic mixture is revised as C50+FA28+MS11+MK11 concrete mix where final total powder content is 910 kg/m³ in which cement content is 455 kg/m³ and pozzolanic mixture is 455 kg/m³.

Table 4 presents several possible binary, ternary and quaternary blended high strength grade (M80) concrete mixes with the quantities of pozzolanic mixtures, their flow properties and achieved strengths. From this table, three optimally blended concrete mixes are selected.

From the experimental investigations, the mixes B2, T3 and Q11 are chosen as optimum binary, ternary and quaternary blended high strength grade (M80) concrete mixes made with fly ash (FA), microsilica (MS) and metakaolin (MK) where both desired workability and strength properties are met along with optimal usage of pozzolanic quantities. The following are mix designations of optimum combinations of binary, ternary and quaternary blended high strength grade (M80) desired mixes:

- (1) C95+MS5 [B2]
- (2) C65+FA20+MS15 [T3]
- (3) C50+FA28+MS11+MK11 [Q11]

Numbers in the above mix designations indicate percentage by weight of total powder content. Total powder content for binary, ternary is 700 kg/m³ and while for quaternary blended high strength grade (M80) it is 910 kg/m³. Thus, by incorporating metakaolin (MK) into micro silica (MS) and fly ash (FA) blended ternary blended desired mixes, the amount of fly ash used has almost doubled to achieve the requisite workability and therefore desired strength. From this observation, it can be understood that micro silica (MS) in blended desired mixtures imparts high strength but flow properties are marginally satisfied while metakaolin (MK) inclusion enhances the usage of high quantity of fly ash in blended concrete mixes for superior rate of gain of strength and more importantly for improved workability of concrete mix. The quaternary blended fly ash based concrete mix made of microsilica (MS) and metakaolin (MK) combination is found to be superior to ternary blended fly ash based concrete mix made with either microsilica (MS) or metakaolin (MK) due to reasons that for similar strength, better early strength, enhanced rate of gain of strength, improved flow properties and more use of fly ash quantity in developing blended high strength grade (M80) concrete.

Table 3. Base Quantities for high strength M80 grade concrete mix

	Ce- ment	Fly ash	Total Powder Content	Fine Aggre- gate	Coarse Aggre- gate	Water (wa- ter/powde r=0.23)
Quan- tity kg/m ³	450	250	700	644	966	150 L

Table 4 presents the summary of all the optimal quantities of binary, ternary and quaternary blended M80 grade concrete mixes. The table also displays the replacement percentages of SCMs, total powder content in kg and water/powder ratios along with corresponding mix numbers and mix designations

In high strength grade (M80), three optimally blended binary and ternary concrete mixes (C95+MS5, C65+FA20+MS15, C50+FA28+MS11+MK11) are chosen based on desired compressive strength achievement. From the studies, it is observed that without inclusion of micro silica (MS), desired high strength cannot be attained. Further investigations have showed that metakaolin (MK) based quaternary blended high strength concrete mix yield better performance than ternary and binary blends in terms of (1) usage of high quantity of fly ash, (2) enhanced fresh properties and (3) reduction in quantity of cement used.

From table 5.34 and Fig 5.4, the total powder content for binary and ternary blended concrete mixes of high strength grade (M80) the total powder content adopted is 700 kg/m³ and whereas for quaternary blended concrete mixes of M80 grade, the total powder

content adopted is 910 (additionally 30% of FA is added). It can be concluded that quaternary blended concrete mixes are more efficient than ternary blended concrete mixes for high strength grade (M80).

Based on the compressive strength attained at specified age of curing, the efficacy of pozzolans are understood. In this study, pozzolans used for blended concrete mixes are Fly Ash (FA), Microsilica (MS) and Metakaolin (MK). Age of curing specified for Fly Ash (FA) blended binary, ternary and quaternary blended concrete mixes of various grades is 60 days while it is 28 days for Microsilica (MS) and Metakaolin (MK) blended concrete mixes.

Metakaolin (MK) blended concrete mixes will set relatively quickly due to its high reactivity, which also prevents bleeding and settling of aggregates. Metakaolin (MK) when compared to micro silica (MS) [10] has similar particle density and surface area but different morphology and surface chemistry. Because of its hydrophilic surface, Metakaolin (MK) is easier to disperse into wet concrete. Metakaolin (MK) can be incorporated at any stage of concrete production; it should be mixed thoroughly to achieve even distribution; intensive mixing is not necessary like micro silica (MS) based concrete

6.2 Studies on Compressive Strength of binary, ternary and quaternary blended concrete mixes

This investigation is carried out to study the compressive strength of binary, ternary and quaternary blended concrete mixes of high strength grade (M80) made with Fly Ash (FA), Microsilica (MS) and Metakaolin (MK) at 3, 7, 28 and 60 days.

Table 6 presents the compressive strength of binary, ternary and quaternary blended concrete mixes of high strength grade (M80) made with Fly Ash (FA), Microsilica (MS) and Metakaolin (MK).

7. Conclusions

Based on the systematic and detailed experimental study conducted on high strength grade (M80) of binary, ternary and quaternary blended concrete mixes made with fly ash (FA), microsilica (MS) and metakaolin (MK) with an aim to develop high performance concrete mixes, the following are the conclusions arrived.

1. Metakaolin blended binary, ternary and quaternary concrete mixes attain early strengths due to its inherent faster reacting capability than microsilica (MS) blended concrete mixes.
2. For development of high strength concrete mixes (M80), use of micro silica is compulsory due to its inherent high reactive property and micro-filler capacity.
3. In development of high strength (M80) grade fly ash blended concrete mixes, both metakaolin and micro silica are required to be added to leverage the benefits of micro-filler capacity of micro silica and early strength attainment of metakaolin. Addition of metakaolin (MK) to blended concrete mixes will enhance early hydration because of its high reactivity.
4. Optimally blended high strength grades M80 quaternary concrete mixes made of should be compacted 50% OPC+28% FA+11% MS+11% MK yields both required workability and desired compressive strengths. From this observation, it can be understood that micro silica (MS) in blended concrete mixtures imparts high strength while metakaolin (MK) inclusion enhances the usage of high quantity of fly ash in concrete mixes for superior rate of gain of strength. So it is evident that both metakaolin and micro silica are required in blended concrete mixes made with low water/powder ratio.
5. From the above observations it can be assumed for better flow and strength realization, in high strength grades (M80) blended fly ash based concrete mixes, the optimum percentage use of metakaolin is found to be 11%.
6. Compressive strengths of metakaolin blended binary, ternary and quaternary concrete mixes have slightly increased than non-

metakaolin blended concrete mixes. Metakaolin cementing reaction rate is very rapid, significantly increasing compressive strength before first three days, which can have various benefits in fast paced construction industry.

7. Metakaolin (MK) is highly reactive alumina silicate whereas micro silica (MS) is reactive silicate. Hence Metakaolin (MK) supplemented concrete mixes have high strengths at all ages because silica and alumina present in Metakaolin (MK) reacts with CH forms CSH (pozzolanic reaction) and CAH (aluminic hydration) respectively which contributes to additional strength than micro silica (MS). So quaternary blended concrete mix made with micro silica (MS) and Metakaolin (MK) has improved microstructure which is dense and impermeable.

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Table 4– Trail mixes of high strength grade (M80) blended concrete mixes

Mix No.	Mix Designation (Values indicate percentage by weight of 'P')	Replacement % (bwp)*				Additional % of FA bwp*	Quantities kg per cu.m				Total Powder Content 'P'	Slump mm	Achieved Strength (MPa)
		OPC	FA	MS	MK		OPC	FA	MS	MK			
C1	C100	100	-	-	-	-	700	0	-	-	700	50	72.35
B1	C65+FA35	65	35	-	-	-	450	250	-	-	700	66	58.94
B2	C95+MS5	95	-	5	-	-	665	-	35	-	700	50	108.56
B3	C90+MS10	90	-	10	-	-	630	-	70	-	700	31	106.04
B4	C85+MS15	85	-	15	-	-	595	-	105	-	700	18	88.32
B5	C95+MK5	95	-	-	5	-	665	-	-	35	700	54	72.15
B6	C90+MK10	90	-	-	10	-	630	-	-	70	700	46	75.78
B7	C85+MK15	85	-	-	15	-	595	-	-	105	700	37	78.82
B8	C80+MK20	80	-	-	20	-	560	-	-	140	700	48	69.35
T1	C65+FA30+MS5	65	30	5	-	-	455	210	35	-	700	55	81.23
T2	C65+FA25+MS10	65	25	10	-	-	455	175	70	-	700	55	94.20
T3	C65+FA20+MS15	65	20	15	-	-	455	140	105	-	700	55	100.54
T4	C65+FA15+MS20	65	15	20	-	-	455	105	140	-	700	46	78.91
T5	C65+FA30+MK5	65	30	-	5	-	455	210	-	35	700	54	76.23
T6	C65+FA25+MK10	65	25	-	10	-	455	175	-	70	700	52	77.34
T7	C65+FA20+MK15	65	20	-	15	-	455	140	-	105	700	51	78.12
T8	C65+FA15+MK20	65	15	-	20	-	455	105	-	140	700	43	67.21
Q1	C65+FA21+MS7+MK7	65	21	7	7	-	455	147	49	49	700	44	90.88
Q2	C60+FA28+MS6+MK6	65	21	7	7	10	455	217	49	49	770	54	82.34
Q3	C54+FA34+MS6+MK6	65	21	7	7	20	455	287	49	49	840	55	72.17
Q4	C65+FA14+MS14+MK7	65	14	14	7	-	455	98	98	49	700	41	80.16
Q5	C59+FA22+MS13+MK6	65	14	14	7	10	455	168	98	49	770	54	81.23
Q6	C54+FA28+MS12+MK6	65	14	14	7	20	455	238	98	49	840	54	83.65
Q7	C50+FA34+MS11+MK5	65	14	14	7	30	455	308	98	49	910	55	71.37
Q8	C65+FA7+MS14+MK14	65	7	14	14	-	455	49	98	98	700	33	90.94
Q9	C58+FA16+MS13+MK13	65	7	14	14	10	455	119	98	98	770	52	93.25
Q10	C53+FA23+MS12+MK12	65	7	14	14	20	455	189	98	98	840	54	94.72
Q11	C50+FA28+MS11+MK11	65	7	14	14	30	455	259	98	98	910	55	110.71
Q12	C46+FA34+MS10+MK10	65	7	14	14	40	455	329	98	98	980	56	79.91

Table 5 - Final optimized mix proportions of blended concrete mixes

Grade of concrete Mix	Mix No	Mix Designation (Values indicate percentage by weight of 'P')	Replacement % (bwp)*				Additional % of FA bwp*	Quantities kg per cu.m				Total Powder Content 'P' kg (i)+(ii)+(iii)+(iv)	Fine Aggregate	Coarse Aggregate	Water	W/P ratio
			OPC	FA	MS	MK		OPC (i)	FA (ii)	MS (iii)	MK (iv)					
M80	B2	C95+MS5	95	-	5	-	-	665	-	35	-	700	644	966	150	0.23
	T3	C65+FA20+MS15	65	20	15	-	-	455	140	105	-	700	644	966	150	0.23
	Q11	C50+FA28+MS11+MK11	65	7	14	14	30	455	259	98	98	910	644	658	150	0.23

bwp* – By weight of Total Powder Content

W/P ratio – Water/Powder Ratio

Table 6 – Compressive Strengths of optimally blended M80 grade concrete mixes

Grade of concrete Mix	Mix No	Mix Designation (Values indicate percentage by weight of Total Powder)	Compressive Strength (MPa)			
			3 days	7 Days	28 days	60 days
M80	B2	C95+MS5	35.43	57.56	108.56	111.22
	T3	C65+FA20+MS15	26.18	50.12	73.17	100.54
	Q11	C50+FA28+MS11+MK11	50.02	64.19	95.26	110.71