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Effect of insoluble residue on properties of Portland cement

Kraiwood Kiattikomol, Chai Jaturapitakkul*, Jatuphon Tangpagasit

Department of Civil Engineering, King Mongkut's University of Technology Thonburi, Bangmod, Tungkru, Bangkok 10140, Thailand

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Abstract

Insoluble residue is a non-cementing material which is present in Portland cement. This residue material affects the properties of cement, especially its compressive strength. To control the non-cementing material in Portland cement, ASTM standard allows the insoluble residue to be not higher than 0.75%. This limitation is much lower than the allowance provided by the British standard which is 1.5%. To verify the effect of insoluble residue on the properties of Portland cement, artificial insoluble residue was prepared and replaced in Portland cement type I. Finely crushed sand was extracted to represent artificial insoluble residue. Setting times and compressive strengths of cement mortar mixed with insoluble residue were investigated. The Portland cement was replaced by insoluble residue which varied in amounts of 0%, 0.5%, 1.0%, 1.5%, 2.0%, 3.0%, 5.0% and 7.0% by weight. The results showed that the addition of the insoluble residue from 0.0% to 7.0% by weight in Portland cement did not affect the normal consistency or setting times of cement. However, the compressive strength of cement mortar was affected during the early age, but the figure reduced as the cement mortar was older. With 7.28% of insoluble residue in the mortar at 1 day, the compressive strength was reduced by 11.5%, but after 60 days, the strength of the same mortar was only reduced by 5.5% as compared to the control mortar. It was also found that the compressive strength of Portland cement mortar with insoluble residue provided by ASTM standard or British standard was still higher than the compressive strength of Portland cement mortar type I allowed by the standards. The limit of insoluble residue given by ASTM standard as 0.75 is rather low and can possibly be increased to 1.5% according to British standard, or even slightly higher, without significantly reducing the compressive strength of cement.

Keywords: Insoluble residue; Portland cement; Mortar; Compressive strength

1. Introduction

Insoluble residue is a measure of adulteration of cement, largely coming from impurities in gypsum and can be found by treating the cement with hydrochloric acid and sodium hydroxide [1]. ASTM C 150 [2] limits the insoluble residue in Portland cement type I not higher than 0.75%. BS 12: 1996 [3] sets the limit for insoluble residue at 1.5% for cement not containing a minor additional constituent, and releases the limit to 5.0% for cement including a minor additional constituent, such as granulated blast furnace slag, natural pozzolana, fly ash or filler [3].

It is found that in modern cement, there is a higher content of C_3S and a greater fineness than that of 40 years ago. As a consequence, cement mortar has, nowadays, a 28 days compressive strength perhaps 25 MPa higher than in 1925 [1]. It seems that a higher insoluble residue in Portland cement can be increased to a higher value without any negative effect on its strength. This premise was confirmed by Poupongphan [4]. They found that with 0.5% of finely crushed brick as an insoluble residue in Portland cement type I, the compressive strength of cement mortar was reduced by 1.6%, and by increasing the

Table 1
Chemical composition of Portland cement type I and finely crushed sand before and after dissolving in acid and basic solution

Oxide (%)	Finely crushed sand before dissolving (%)	Finely crushed sand after dissolving (%)	Portland cement type I (%)
SiO_2	88.94	91.27	20.2
Al_2O_3	5.09	5.07	5.42
Fe_2O_3	1.74	0.41	2.92
CaO	2.79	0.09	63.82
K_2O	2.79	2.83	0.9
TiO_2	0.14	0.15	–
Insoluble residue (%)	92.81	100.0	0.28

* Corresponding author. Tel.: +66-2-470-9133; fax: +66-2-427-9063.

E-mail address: ichakkul@cc.kmutt.ac.th (C. Jaturapitakkul).

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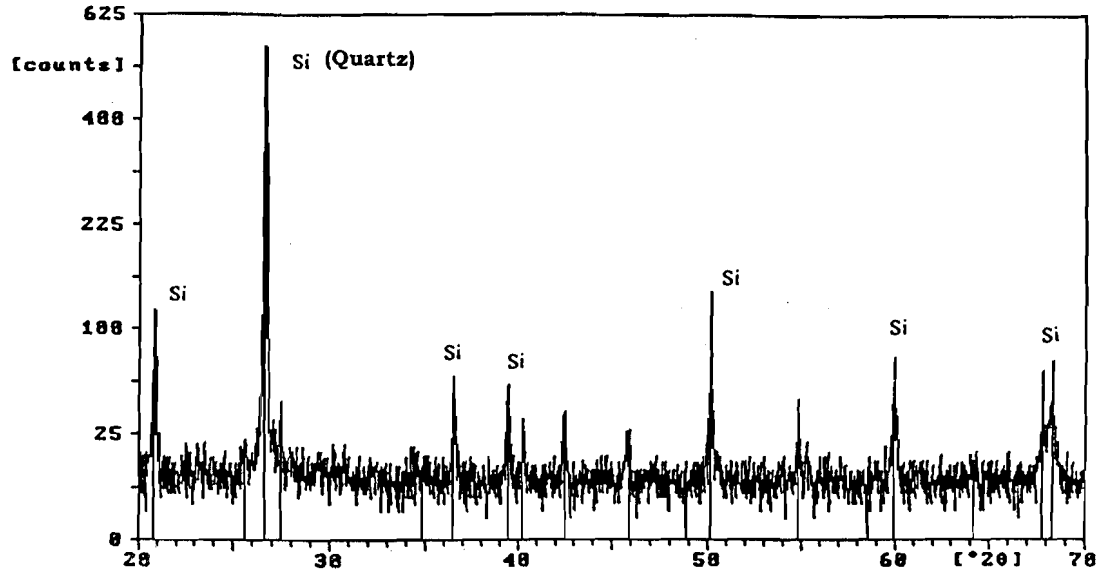

Fig. 1. The XRD of finely crushed sand after dissolving in acid and basic solution.

insoluble residue up to 1.5%, this mixture resulted in a lowering of the cement mortar strength of less than 4% compared to the control cement mortar strength at 28 days. Normal consistency and setting times are not changed by the addition of insoluble residue in cement. However, this premise still needs more data for support and confirmation that slightly increased rates of insoluble residue are not a major factor affecting its strength.

2. Objective

The objective of this study is to investigate the effect of insoluble residue on the properties of Portland cement. Finely crushed sand was used as the insoluble residue. The particle size distributions of the finely crushed sand and the Portland cement were tested and compared. Normal consistency and setting times of the Portland cement mixed with insoluble residue were evaluated and compared to the cement paste. The compressive strength of cement mortar, with and without the insoluble residue, was also investigated. The results will provide cement researchers an understanding of what happens to the Portland cement given the amount of insoluble residue provided by the cement standards.

3. Experimental program

In this experiment, river sand was used as an insoluble residue. The artificial insoluble residue was prepared as follows. First, the river sand was crushed by using a ball mill and then sieved through a no. 200 sieve. Second, the fine powder was mixed with water and dissolved in hydrochloric acid followed, after filtration, by further digestion in sodium hydroxide. An ammonium nitrate solution was then used in the final washing. To prevent finely ground insoluble residue from passing through the filter paper, medium-textured paper was used as a filter. The procedure to obtain the artificial insoluble residue material followed ASTM C 114 [5] except that the

Table 2
Added and tested results of the insoluble residue in cement

Sample number	Added insoluble material (%)	Total insoluble material in cement (%)		Difference (%)
		Calculation	Test	
A00	0.0	0.28	0.28	0.0
B05	0.5	0.78	0.54	0.24
C10	1.0	1.28	1.04	0.24
D15	1.5	1.78	1.50	0.28
E20	2.0	2.28	2.14	0.14
F30	3.0	3.28	3.04	0.24
G50	5.0	5.28	4.96	0.32
H70	7.0	7.28	6.86	0.42

Table 3
Results of normal consistency and setting times of cement pastes mixed with insoluble residue

Sample number	Insoluble residue (%)	Normal consistency (%)	Setting times (min)	
			Initial	Final
A00	0.28	26.8	108	195
B05	0.78	26.8	108	210
C10	1.28	26.8	109	195
D15	1.78	26.8	111	210
E20	2.28	26.8	109	210
F30	3.28	26.8	109	195
G50	5.28	26.8	105	210
H70	7.28	26.8	102	195

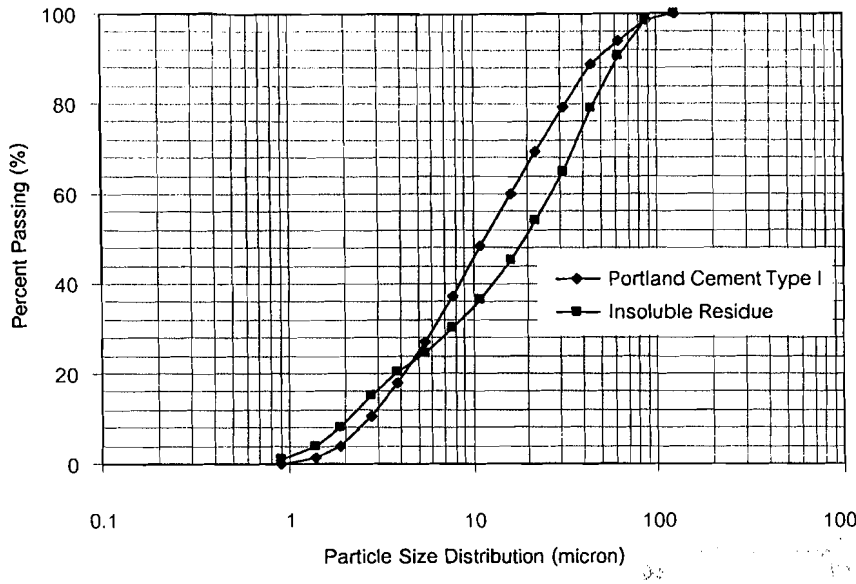

Fig. 2. Particle size distribution of cement and insoluble residue.

concentrations of the acid and the basic solutions were two times higher than the concentration given by the standard. This guaranteed that the obtained material was not dissolved in acid or basic solution. Chemical compositions between insoluble residue of the finely crushed sand and Portland cement were then compared.

Portland cement type I was replaced with the insoluble residue by 0%, 0.5%, 1.0%, 1.5%, 2.0%, 3.0%, 5.0%, and 7.0% by weight of cement, plus the existing insoluble residue in cement. The mixed cement specimens were tested to confirm their amount of insoluble residue using the ASTM C 778 [6] standard. Normal consistency and setting times of the mixed cement were tested in accordance to ASTM C 187 [7] and ASTM C 191 [8], respectively. The mixed cement was also used to prepare mortar in according to ASTM C 109 [9]. After 24 h, the molds were removed and all the 5-cm cube specimens were cured in saturated lime water. The compressive strengths of mixed cement mortar were tested at the age of 1, 3, 7, 14, 28, and 60 days. At each date of testing, the data are the average of seven specimens.

4. Results and discussion

4.1. Chemical composition of insoluble residue

X-ray fluorescence spectrometry was used to test the chemical composition of Portland cement type I and finely crushed sand, before and after dissolving in hydrochloric acid and sodium hydroxide. The results are shown in Table 1. Portland cement and finely crushed sand, after dissolving in the acid and basic solution, consist of the insoluble residue 0.28% and 100%, respectively. The major oxide of the residue material is silicon dioxide which is 91.27% of the total weight, while calcium oxide is the major oxide of Portland cement. It is noted that the oxides of the finely crushed sand before and after dissolving in hydrochloric

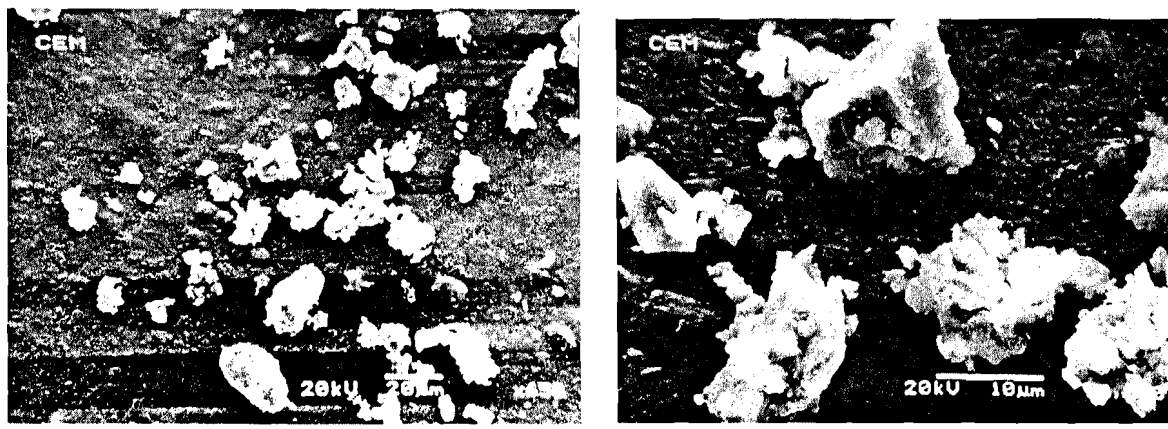

Fig. 3. Scanning electron microscopy of cement.

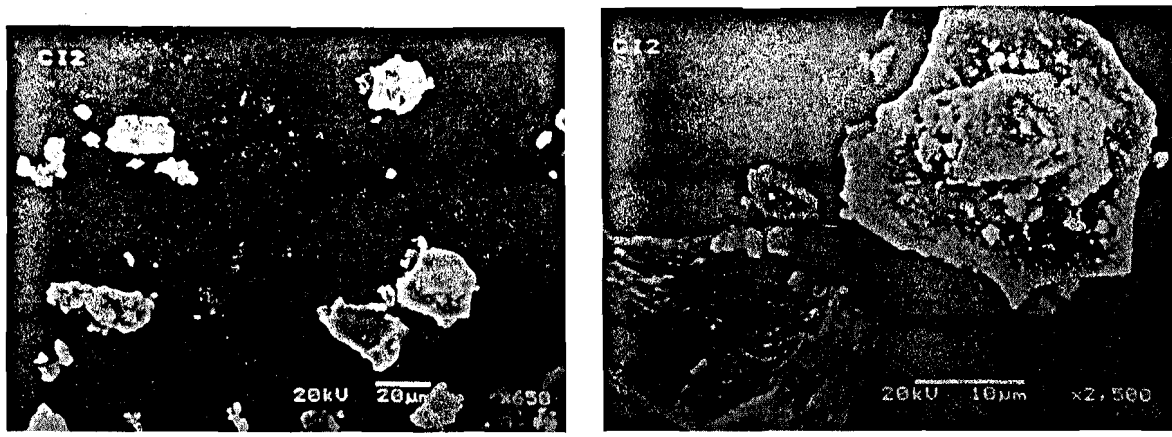

Fig. 4. Scanning electron microscopy of insoluble residue.

acid and sodium hydroxide are almost the same except that the CaO and Fe_2O_3 contents were reduced from 2.79% and 1.74%, respectively, to 0.09% and 0.41%, while SiO_2 is increased from 88.94% to 91.27%. Finely crushed sand, before and after dissolving, has little difference in chemical composition.

Fig. 1 showed the result of chemical composition analyzed by X-ray diffraction (XRD). The graph shows the crystalline phase and the amorphous phase of insoluble residue. The quartz content in the sand is very high and similar to the analysis result by X-ray fluorescence spectrometry. XRD analysis reveals that quartz is in the form of the crystalline phase. This means that the finely crushed sand after dissolving in acid and basic solution can be used as an insoluble material which does not react with cement.

4.2. Insoluble residue in Portland cement and in the replaced cement

After the insoluble material was replaced in cement at the proposed percentage, the cement and insoluble residues were mixed together to make the sample uniform. Then, the percentage of insoluble residue in the mix was checked. The proposed and the tested results of the insoluble residue material in the mix are shown in Table 2. It is seen that the insoluble residue of cement (sample A00) is very low; it is 0.28% which is lower than the limit given by the ASTM (0.75%) and BS (1.5%) standards. All of the test results show the lower percentage of insoluble residue compared to the calculated insoluble residue. The difference of insoluble residue ranges from 0.24% in sample B05 (0.5%) to 0.42% in sample H70 (7.0%). However, these figures are still very close to the proposed values.

4.3. Effect of insoluble residue on normal consistency and setting time of Portland cement

The effect of insoluble residue for each percentage as a cement replacement on normal consistency and setting times were presented in Table 3. The replacement of insoluble residue up to 7.0% did not change the normal consistency of cement. The required water for normal consistency was still the same and remains at 26.8%. This is due to the same particle size distribution of the insoluble residue and the cement. Fig. 2 shows the particle size distribution of two materials determined by Particle Analyzer Microtrac II. Since both materials had almost the same particle size

Table 4
Compressive strength of cement mortar containing insoluble residue

Sample number	Insoluble residue (%)	Compressive strength (MPa)					
		1 day	3 days	7 days	14 days	28 days	60 days
MA00	0.28	18.2	29.6	36.5	39.9	45.3	49.2
MB05	0.78	17.8	29.0	36.2	39.5	44.6	48.6
MC10	1.28	17.4	28.7	35.7	39.0	44.4	47.9
MD15	1.78	17.2	28.3	35.6	38.7	43.9	47.9
ME20	2.28	16.9	28.0	35.0	38.3	43.3	47.3
MF30	3.28	16.7	27.5	34.4	38.2	43.1	47.1
MG50	5.28	16.3	26.9	34.2	37.8	42.6	46.9
MH70	7.28	16.1	26.8	34.0	37.0	42.3	46.5

Table 5
Percentage of compressive strength of cement mortar containing insoluble residue comparing to the control mortar

Sample number	Insoluble residue (%)	Percentage of compressive strength (%)					
		1 day	3 days	7 days	14 days	28 days	60 days
MA00	0.28	100.0	100.0	100.0	100.0	100.0	100.0
MB05	0.78	97.8	98.0	99.2	99.0	98.5	98.8
MC10	1.28	95.6	97.0	97.8	97.7	98.0	97.4
MD15	1.78	94.5	95.6	97.5	97.0	96.9	97.4
ME20	2.28	92.9	94.6	95.9	96.0	95.6	96.1
MF30	3.28	91.8	92.9	94.2	95.7	95.1	95.7
MG50	5.28	89.6	90.9	93.7	94.7	94.0	95.3
MH70	7.28	88.5	90.5	93.2	92.7	93.4	94.5

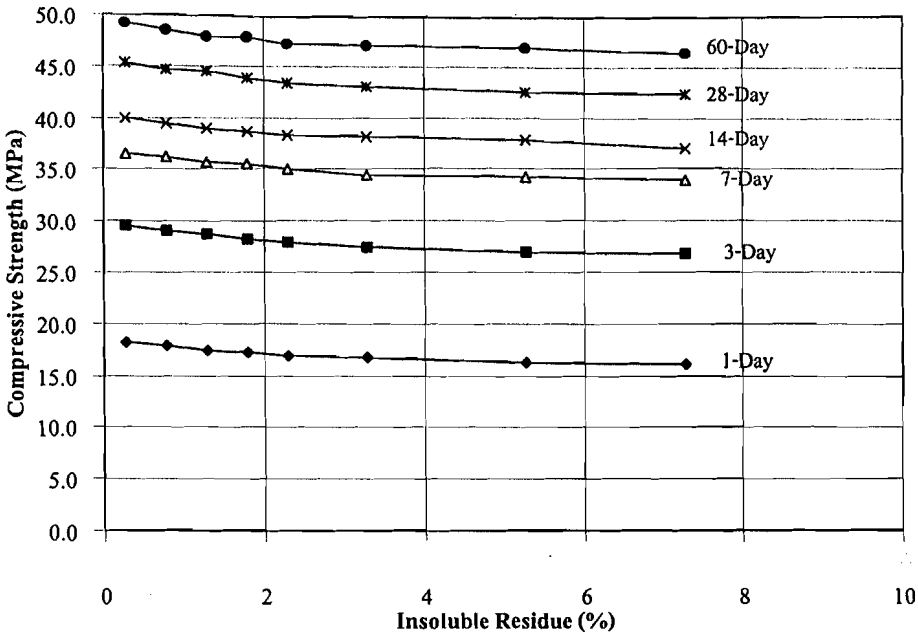

Fig. 5. Relationship between compressive strength of cement mortar and percent of insoluble residue.

distribution, they needed the same amount of water to maintain the same normal consistency.

Figs. 3 and 4 show pictures of cement and insoluble residue taken by scanning electron microscopy. Both cement and insoluble residues have an irregular, angular and crushed shaped with no spheres. These pictures confirm that the insoluble residue has little effect of normal consistency since cement and insoluble residue had the same shaped particles.

Initial and final setting times of sample A00 (only cement paste) were 108 and 195 min, respectively. With the replacement of cement by insoluble residue up to 7.0%, the setting times were changed little; they varied from 102 to 111 min for the initial setting time and from 195 to 210 min for the final setting time. This means that there is no effect of insoluble residue on setting time of cement paste by replacing insoluble residue up to 7.0%.

4.4. Effect on compressive strength

The compressive strengths of cement mortar containing insoluble residue up to 7.28% at various ages are shown

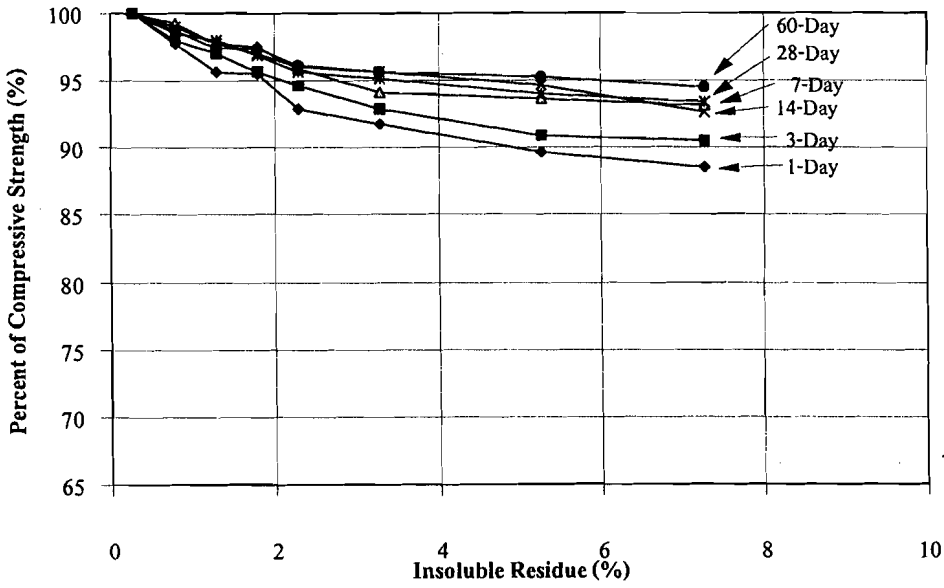

Fig. 6. Relationship between percentage compressive strength of cement mortar and replacement of insoluble residue in the mix.

in Table 4. Table 5 shows the percentage of compressive strength of cement mortar mixed with insoluble residue compared to the control (sample MA00) mortar strength. Fig. 5 shows the relationship between the compressive strength of cement mortar and percentage of insoluble residue in the mix. Fig. 6 shows the relationship between the percentage of compressive strength of cement mortar and percentage of insoluble residue.

It is seen that the higher the percentage is of insoluble residue in cement mortar, the lower the compressive strength becomes. The compressive strengths of control samples vary from 18.2 MPa at 1 day to 49.2 MPa at 60 days. The sample with 0.5% replacement of insoluble residue has the compressive strengths of 17.8 MPa at 1 day and increases to 48.6 MPa at 60 days. These strengths are lower than the control strength 2.2% at 1 day and 1.2% at 60 days, respectively. The compressive strengths of cement mortar with 1.5% of insoluble residue, sample MD15, are, respectively, 17.2 MPa at 1 day and 47.9 MPa at 60 days. They are lower than the control strength — about 5.5% at 1 day and 2.6% at 60 days. It is noted that the reduction of strength due to insoluble residue is rather high at the early ages and tends to reduce when the age of cement mortar increases.

With the highest amount of insoluble residue in the mix, 7.28%, in sample MH70, it is found that the compressive strength is still higher than the limit given by ASTM C 150 [2]. For ASTM C 150 [2], the cement mortar strength with 0.75% of insoluble residue has to be not lower than 12.4 MPa at 3 days and 19.3 MPa at 7 days for Portland cement type I. Sample MH70 gives compressive strength of mortar at 3, 7 and 28 days equal to 26.8, 34.0 and 42.3 MPa, respectively. This means that the insoluble residue up to 7.28% in cement is not seriously harmful to its strength. It reduces the strength of cement mortar, but it is not the main factor affecting the strength of cement mortar. The value of the insoluble residue limited by ASTM C 150 [2], 0.75%, seems to be rather low and can be slightly increased without lowering the standard of cement.

5. Conclusions

From the results of these experiments, the following conclusions can be drawn.

(1) The major constituent of the insoluble residue used in this research was silicon dioxide (quartz) derived from acid-treated river sand. Quartz is normally recognised to be a non-cementing material.

(2) The normal consistency and setting times of cement were not significantly affected by finely divided sand at cement insoluble residue material levels up to and including 7.28%. This may be because the particle size distribution and the shape of the insoluble residue were similar to that of the Portland cement.

(3) The compressive strength of ASTM C 109 mortar cubes reduced progressively as the insoluble residue was increased. The strength reduction was greater at 1 and 3 days than at test ages from 7 to 60 days.

(4) The limit of insoluble residue given by ASTM C 150 at 0.75% may be rather low and could possibly be increased to 1.5% (as in BS 12: 1996), or even slightly higher without significantly reducing the compressive strength of cement mortar.

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