Research Article

Enhancement of hardness in nanostructured CuO/TiO_2–cement composites

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Abstract

Concrete has been widely used in pavements and buildings, and it is necessary to increase its hardness in order to resist deformation, penetration, and abrasion for these applications. This study explores the effects of addition of CuO and/or TiO_2 nanoparticles on the hardness of cement mortar. Scanning electron microscopy, X-ray diffraction, energy dispersive spectroscopy, and Rockwell hardness testing were used to study the microstructure, chemical and phase composition, and hardness. The results showed that the addition of nanoparticles can effectively improve the hardness of cement mortar by improving the microstructure and hydration process. This is because they offer additional nucleation sites for hydrates to deposit on, producing a more compact microstructure with finer grains. As the concentration of nanoparticles added increases, the enhancement is more obvious and stable. However, after the concentration reaches its maximum level, the hardness starts to decrease due to the formation of defects, mainly voids, caused by excess nanoparticles. CuO and TiO_2 nanoparticles show similar effects on the microstructure and hardness, but the addition of both CuO and TiO_2 nanoparticles with similar amounts shows denser microstructure and higher hardness as they densify the composites.

Keywords Cement mortar · CuO · TiO_2 · Nanoparticles · Hardness

1 Introduction

Concrete is one of the most important and commonly used building materials in modern constructions. The hardness of concrete decides its ability to resist deformation, abrasion, and penetration. Usually, the higher the hardness of the concrete, the better the surface quality of the structures and pavements made from concrete.

Because of the unique properties of composite materials, studies on polymer composite [1], carbon fiber composite [2], sandwich composite structures [3], and addition of nanoparticles [4] have been considered as effective means to modify the mechanical properties of conventional construction materials.

Nanotechnology holds great potential for solving many common problems in building materials, such as cracking and chemical attacks [4]. Currently, nanoconcrete, which is generated by adding nanoparticles with particle size less than 500 nm into Portland cement, has been proven to be a greener and stronger material compared to traditional concrete [5]. Nanoparticles are fillers modifying concrete's microstructure and promoting hydration as catalyst with high reactivity, thus improving the durability of concrete [6]. Nair et al. found that TiO_2 and CuO nanoparticles could strengthen concrete, and the composites could be more resistant to saline and acidic corrosion [7]. Jo et al. concluded that SiO_2 nanoparticles could improve the strength of cement mortar by filling the structural voids and promoting the pozzolanic reaction [8]. The abrasion resistance

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and compressive strength were both found to increase with the increase in the percentages of added CuO and SiO_2 nanoparticles by Riahi et al. [9]. Li et al. reported the increase in both short-term and long-term concrete strength by the addition of SiO_2 nanoparticles [10]. Nazari et al. found that Al_2O_3 nanoparticles increased the compressive strength of concrete, but reduced its workability [11]. Li et al. observed that Fe_2O_3 and SiO_2 nanoparticles increased the compressive and flexural strengths of cement mortar by changing its microstructure [12].

Most of the previous research focused on the effect of a single kind of nanoparticles, and only a few papers have been found on the combined effect of two kinds of nanomaterials. Ahmed et al. [13] found that by adding both multiwall carbon nanotubes (MWCNTs) and $MnFe_2O_4$ nanoparticles, the concrete achieved an improvement in compressive and flexural strength by 19% and 21%, respectively, compared to the strength of plain concrete and those with only MWCNTs or $MnFe_2O_4$ nanoparticles [14]. Li et al. [15] reported that under standard curing process, the addition of an optimal combination of silica and limestone nanoparticles increased both the flexural and compressive strength compared with the addition of silica or limestone nanoparticles, respectively.

These aforementioned studies show that addition of nanoparticles improves strength. As the hardness of concrete usually increases with the increase in its compressive strength [16], it would be reasonable to assume that nanoconcrete would be harder than traditional concrete and thus more resistant to abrasion, a feature generally determined by the hardness [17]. Li et al. reported that pavement concrete incorporated with TiO_2 and SiO_2 nanoparticles increased the abrasion resistance [16], and Ardalan et al. showed that adding SiO_2 nanoparticles accelerated the curing process [17]. Niewiadomski et al. [18] reported nanohardness enhancement by adding SiO_2 or TiO_2 or Al_2O_3 nanoparticles, and León et al. [19] found that SiO_2 or Al_2O_3 nanoparticles increased the surface hardness of the concrete. On the contrary, León [19] found that the addition of a combination of SiO_2 and Al_2O_3 nanoparticles did not increase the hardness at all. As we see, most of the existing research focuses on the effect of single kind of nanoparticles, although some studies have explored the combined effect of two kinds of nanomaterials. Nevertheless, the results reported are controversial. Therefore, it is necessary to conduct further studies on using nanoparticles to improve the hardness of concrete.

Based on literature review, TiO_2 is the most widely used material for enhancing the properties of concrete. It can improve both the tensile and flexural strength of concrete [20] and facilitate the hydration reaction of cement at the early age [21]. Moreover, concrete with TiO_2 nanoparticles can clean organic pollutants on the surface due to its photo-catalytic property [22]. On the contrary, only a few studies have been done using CuO nanoparticles. CuO nanoparticles were found to increase the tensile strength of concrete by forming more hydrates [23] and to reduce the water absorption and chloride permeability of cement mortar [24]. Besides, the combination of CuO nanoparticles and fly ash was found to be able to improve the electrical resistivity and durability of the concrete [25]. These results show that CuO and TiO_2 nanoparticles can not only effectively improve the mechanical properties of concrete but also make additional benefits. Therefore, they are chosen for this study.

In this research, we systematically studied the effects of CuO, TiO_2, and the combination of TiO_2 and CuO nanoparticles on the microstructure and mechanical properties of concrete. The results show that the nanoparticles can effectively increase the hardness of composites by densifying the microstructure.

2 Experimental

2.1 Materials

QUIKRETE® Concrete Mix (No. 1101) produced by QUIKRETE Inc. in USA was used as the matrix material. It is a mixture of Portland cement and aggregates of sand and gravel. CuO and TiO_2 nanoparticles were provided by Guangdong Mitake Company in China with a purity of 99.9 wt%. Local (Saskatoon, SK, Canada) tap water with total hardness of 182 mg $CaCO_3$/L was used.

2.2 Sample preparation

Rockwell indentation testing was chosen for hardness measurement in this work, which was conducted on the samples using an Instron Wolpert GmbH (Ludwigshafen, Germany) 751 tester. A load of 10 kgf (98 N) and a 1/16 inch steel ball HRB indenter were utilized. For each sample, the hardness value was averaged from 20 measurements conducted on the sample surface. The testing procedures follow ASTM E18. Considering the indenter size, in order to avoid testing the hardness on gravels, the concrete mortar was first passed through a sieve with a diameter of 2 mm to remove the large gravels. Then, the sieved mortar was put into a large beaker to mix with water by continuous stirring for 15 min to obtain a mixture. Nanoparticles were then added to the mixture by continuous stirring to make a nanocomposite mixture, which was then poured into a mould with vibration to form a smooth horizontal surface. The surface was covered with a thin plastic sheet to keep moisture for 24 h. After that, the samples were demolded and kept under clear tap water at a room temperature of

23 ℃ for 28 days before hardness tests. Water-to-solid-mixture ratio of 0.2 mL/g was chosen based on a pre-hardness testing as it generated the highest hardness value and showed good fluidity without obvious stratification during the sample-making process.

In total, 37 concrete samples with dimensions of $3\times1.5\times1\ cm^3$ were prepared, which include 36 composite samples with the addition of nanopowders and one plain concrete sample as reference. The 36 nanoconcrete samples were divided into six groups with different weight ratios of added nanoparticles to the cement mortar. The percentages of nanoparticles from group 1 to 6 is 1 wt%, 3 wt%, 5 wt%, 7 wt%, 10 wt%, and 15 wt%, respectively. Within each group, there are six subgroups in which TiO_2/CuO proportions were 20:80, 40:60, 60:40, 80:20, 0:100, and 100:0, respectively. The compositions of the samples are shown in Table 1. In the table, NCT1-1 represents subgroup 1 of group 1, which indicates a sample having 1 wt% of the cement mortar replaced by nanoparticles with 20 wt% TiO_2 and 80 wt% CuO. The rest of the samples were named in similar way. For the first four samples in group 1, 15 g

Table 1 Composition of nanoconcrete samples

Sample name	Cement mix (g)	Water (mL)	Nano-TiO_2 (g)	Nano-CuO (g)	Total percentage of cement mix (%)
NCT1-1	30	6	0.06	0.24	99
NCT1-2	30	6	0.12	0.18	99
NCT1-3	30	6	0.18	0.12	99
NCT1-4	30	6	0.24	0.06	99
NCT1-5	15	3	0.15	0	99
NCT1-6	15	3	0	0.15	99
NCT2-1	15	3	0.09	0.37	97
NCT2-2	15	3	0.19	0.27	97
NCT2-3	15	3	0.28	0.18	97
NCT2-4	15	3	0.37	0.09	97
NCT2-5	15	3	0.46	0	97
NCT2-6	15	3	0	0.46	97
NCT3-1	15	3	0.16	0.63	95
NCT3-2	15	3	0.32	0.47	95
NCT3-3	15	3	0.47	0.32	95
NCT3-4	15	3	0.63	0.16	95
NCT3-5	15	3	0.79	0	95
NCT3-6	15	3	0	0.79	95
NCT4-1	15	3	0.23	0.9	93
NCT4-2	15	3	0.45	0.68	93
NCT4-3	15	3	0.68	0.45	93
NCT4-4	15	3	0.9	0.23	93
NCT4-5	15	3	1.13	0	93
NCT4-6	15	3	0	1.13	93
NCT5-1	15	3	0.33	1.34	90
NCT5-2	15	3	0.67	1	90
NCT5-3	15	3	1	0.67	90
NCT5-4	15	3	1.33	0.34	90
NCT5-5	15	3	1.67	0	90
NCT5-6	15	3	0	1.67	90
NCT6-1	15	3	0.53	2.12	85
NCT6-2	15	3	1.06	1.59	85
NCT6-3	15	3	1.59	1.06	85
NCT6-4	15	3	2.12	0.53	85
NCT6-5	15	3	2.65	0	85
NCT6-6	15	3	0	2.65	85
Control	15	3	0	0	100

more concrete mortar was used in order to weight nanoparticles more accurately.

2.3 Material characterization

The structural properties of the obtained samples were investigated by high-resolution field emission scanning electron microscopy (Hitachi SU-8010 SEM), energy dispersive X-ray spectroscopy (EDS), and X-ray diffraction (Rigaku Ultima IV XRD). The average grain size and size distributions of the nanoparticles were generated by processing the SEM images. A random straight line was drawn through the SEM image. The number of grain boundaries intersecting the line was counted. The average grain size was calculated by dividing the number of intersections by the actual line length. The number of grains in each size range was then counted through the image and divided by the number of total grains to generate its fraction. Samples NCT6-6 and NCT6-5 were chosen for EDS testing since they have the highest weight percentage of nanoparticles added, which give a more obvious and typical representation of the element distribution of all the samples. For SEM testing, in addition to samples NCT6-6 and NCT6-5, to test the samples with mixed nanoparticles, NCT5-3 was chosen to represent the samples with high addition amount as it gives the highest hardness. NCT2-3 was chosen to represent the samples with low addition amount.

3 Results and discussion

3.1 SEM analysis for the nanoparticles

Figure 1 shows the SEM morphologies of CuO and TiO_2 nanoparticles. Both of them are mainly in sphere or capsule shapes. The size distributions of the nanoparticles were calculated and are shown in Fig. 2. The average diameters of CuO and TiO_2 nanoparticles are 67.3 and 121.9 nm, respectively. One can see that the size distribution of TiO_2 nanoparticles is more uniform compared to that of CuO. CuO has a smaller crystal size and thus higher relative surface area and higher surface energy, which may result in the generation of agglomerations and produce an uneven distribution.

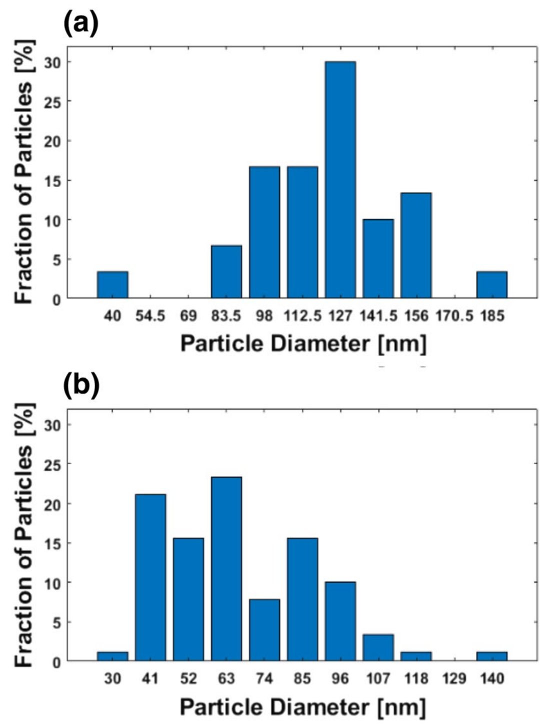

Fig. 2 Size distribution for **a** TiO_2 and **b** CuO nanoparticles

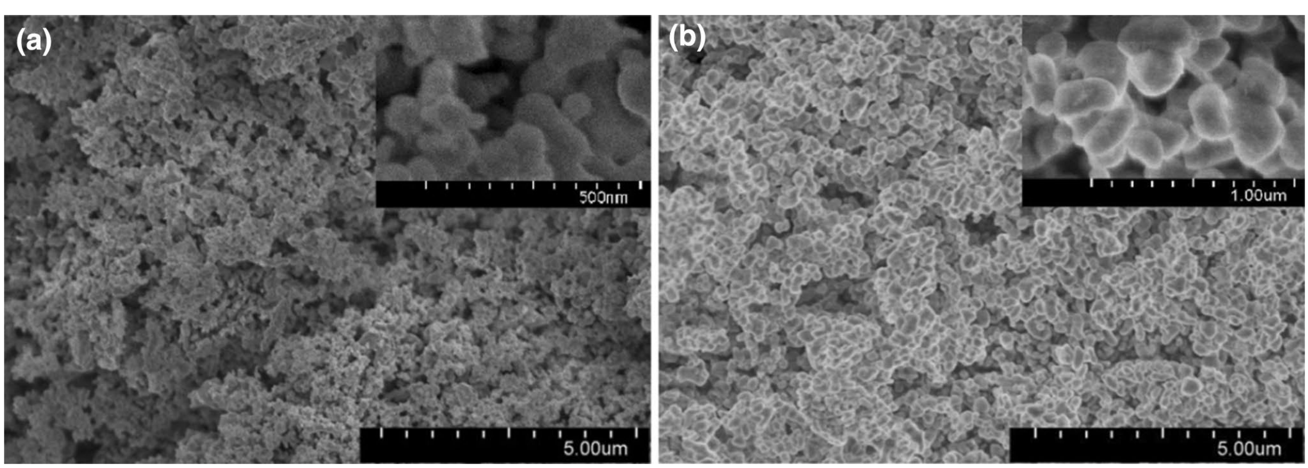

Fig. 1 SEM morphologies of the nanoparticles **a** CuO and **b** TiO_2

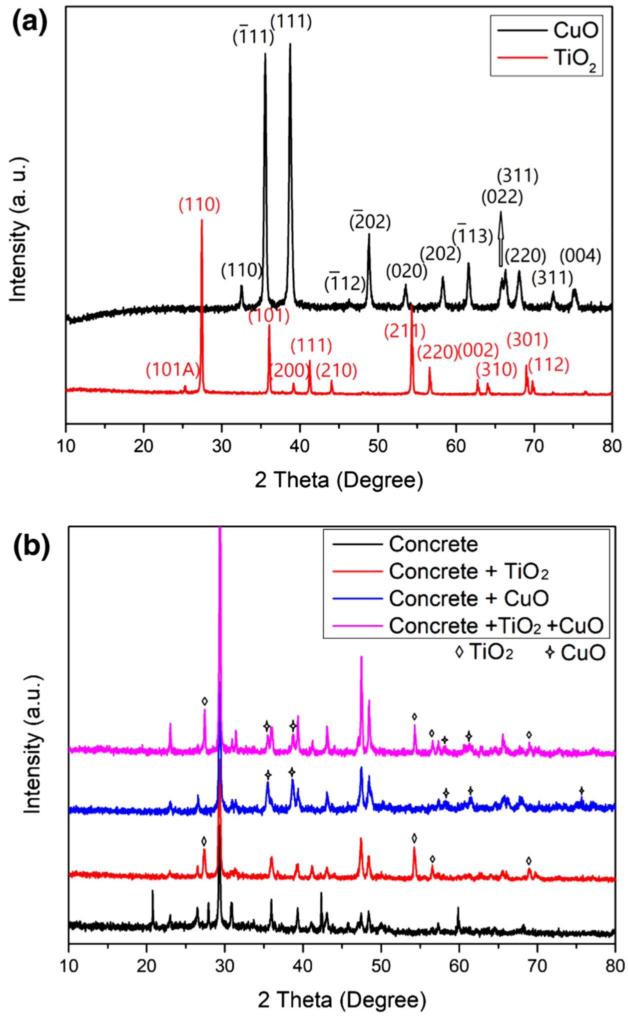

Fig. 3 X-ray diffraction patterns of **a** CuO and TiO_2 nanoparticles, and **b** plain concrete, TiO_2–concrete composite, and CuO–concrete composite, and TiO_2–CuO–concrete composite

3.2 XRD analysis

XRD results of the nanoparticles are illustrated in Fig. 3. CuO shows a single monoclinic structure [26], but TiO_2 displays a mixture of rutile and anatase phases [27]. According to the semi-quantitative XRD analysis proposed by Copeland and Bragg [28], the ratio of the integrated intensities of the XRD peaks of the two components in a mixture is proportional to the ratio of the weight fractions. We can see that only one peak (101) from anatase phase can be identified with very low intensity, indicating that rutile phase is the main component. Figure 3b shows the XRD results of the concrete samples. The strongest peak at $2\theta = 29.9°$, which can be found in all samples, is a typical XRD peak for concrete [29, 30]. For the nanocomposite samples, CuO or rutile TiO_2 peaks appear in addition to the cement peaks. Namely, the peaks at $2\theta = 27°$, 54.5°, 56.3°, and 69° for the composite samples with addition of TiO_2 confirm the presence of TiO_2, and the peaks at $2\theta = 57.5°$, 61.5°, and 71° for the composite samples with addition of CuO confirm the existence of CuO. In the composite concrete sample with mixed nanoparticles added, both CuO and TiO_2 peaks can be found.

3.3 EDS Analysis

EDS area scanning graphs are shown in Fig. 4, where Ti is marked in green, while Cu is marked in red. For samples with added CuO nanoparticles, as shown in Fig. 4a, CuO is observed and dispersed with local agglomerations. For samples with added TiO_2 nanoparticles (see Fig. 4b), Ti is observed and uniformly distributed. Large round dark areas shown in the images are probably silica sand.

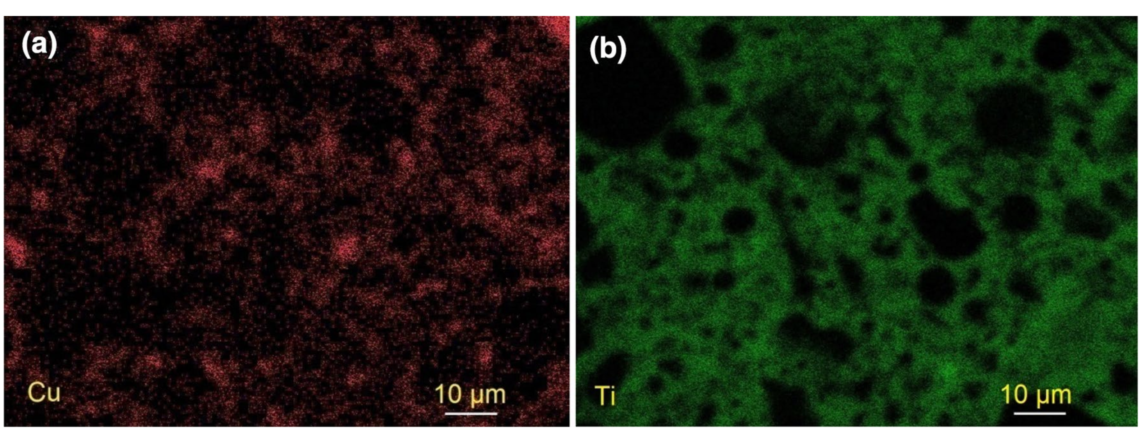

Fig. 4 EDS of **a** Cu in CuO–concrete composite (NCT6-6), and **b** Ti in TiO_2–concrete composite (NCT6-5)

3.4 SEM results

Figure 5 shows the surface microstructures of the plain concrete, concrete with added CuO or TiO_2 nanoparticles, and two samples with addition of both CuO and TiO_2 nanoparticles.

As shown in Fig. 5a, plain concrete shows relatively large grains of irregular shapes. The surface is rough with large voids. With the addition of either TiO_2 (Fig. 5b) or CuO (Fig. 5c), the microstructures are much denser, and grains are much finer. Small sphere-like grains of similar sizes are uniformly distributed on the surface, forming a compact and smooth surface. The voids and porosity are reduced, which is consistent with a previous report [8].

With the addition of small amounts of both nanoparticles (1.2 wt% of nano-CuO and 1.8 wt% of nano-TiO_2), the microstructure of the nanoconcrete (Fig. 5d) is partially improved compared to the plain concrete, and with the addition of 10 wt% of both the nanoparticles (4 wt% of nano-CuO and 6 wt% of nano-TiO_2), the sample shows very dense and fine grain structure, as shown in Fig. 5e. The grain size distribution is uniform, and the surface is very smooth. The structure is improved compared with the concrete with a single kind of nanoparticles added.

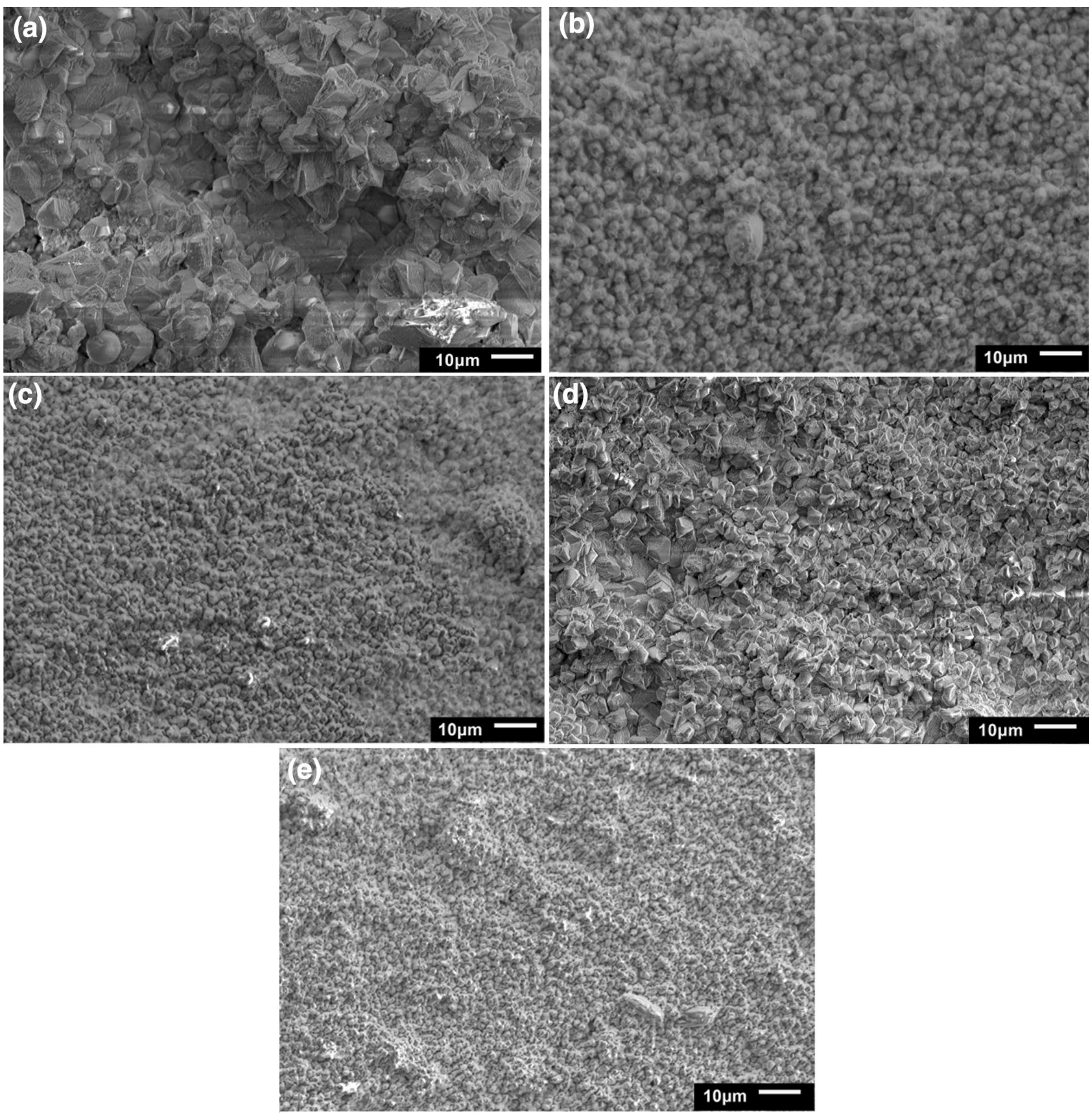

Fig. 5 SEM of **a** plain concrete, **b** concrete with TiO_2 addition (NCT6-5), **c** concrete with CuO addition (NCT6-6), **d** concrete with both nanoparticles under low addition amount (NCT2-3), and **e** concrete with both nanoparticles under high addition amount (NCT5-3)

3.5 Rockwell hardness analysis

Table 2 shows the Rockwell hardness test results for all the samples, and the values presented are the average of 20 measurements with standard deviation. The average hardness of the control group is 79.9 HRB.

In total, 81% of the composite samples show enhancement in hardness compared to the control group, the increment ranging from 0.4% to 8.1%. Sample NCT5-3 (4 wt% nano-CuO and 6 wt% nano-TiO_2) shows the highest hardness of 86.4 HRB. 19% of the samples show decreased or unchanged hardness, and all belong to the first three groups with relatively low percentages of added nanoparticles.

In terms of group averages, values of groups 1 and 2 are similar to that of control group. The average hardness increases from group 2 to group 5, reaching a peak value of 84.5 HRB for group 5, and group 6 shows a reduced value of 83.3 HRB.

These results indicate that the addition of a small percentage of nanoparticles (1 wt% and 3 wt%) is insufficient to modify the microstructure of the concrete, as shown in Fig. 5d for NCT2-3, and thus, no enhancement of hardness appears. When the added nanoparticles range from 5 wt% to 10 wt%, the hardness increases as the percentage of added nanoparticles increases. This can be explained from their modified microstructures as shown in Fig. 5. The added nanoparticles promote the hydration process and thus decrease the grain size and densify the structure, consistent with the results reported by Miyandehi et al. [31]. Research shows that both CuO [32] and TiO_2 [33] are able to promote cement hydration reaction due to their high reactivity. Especially, they offer additional nucleation sites for hydrates to deposit on, which refines grains and thus increases surface-area–to-volume ratio [34]. The nanoparticles act as small kernels attracting abundant C–S–H hydrates surrounding them to form small aggregates in concrete, which facilitates a microstructure with finer grains and more compacted structure [34]. Moreover, the nanoparticles facilitate the growth of more homogenous $Ca(OH)_2$ crystals [9]. Furthermore, the C–S–H gel is in nanoscale and form a coherent structural framework between the nanoparticles and the hydrates [32]. Previous reports found that for TiO_2 nanoparticles, hydration of rutile surfaces is more strongly exothermic than the hydration of anatase surfaces [35, 36], which is consistent with this work. In this work, as shown in Fig. 3a, the main component is rutile phase in TiO_2 nanoparticles. As a result, a finer, more coherent, and more intimately connected internal structure as shown in Fig. 5b, c, e with higher hardness is generated. However, for group 6, extra added nanoparticles reduce the distance between the nanoparticles, which restricts the normal formation of $Ca(OH)_2$ and therefore forming coarse and porous microstructure [37]. Furthermore, excess nanoparticles that cannot be incorporated in the hydration process may cause an inappropriate distribution of nanoparticles, which results in weak zones that lower the mechanical properties [38].

Figure 6 shows the Rockwell hardness results within each group. For group 1 to 4, the highest hardness within each group is in the sample added with a single kind of nanoparticles, but for groups 5 and 6, the samples with a combination of CuO and TiO_2 offer the highest hardness. One of them provides the highest hardness among the 37 samples.

In terms of subgroup 1 to 4 with combined nanoparticles, in groups 1 and 2, the samples with a ratio of 80 wt% CuO and 20 wt% TiO_2 show the highest hardness. From group 3 to 6, the ratio with 60 wt% TiO_2 and 40 wt% CuO reaches the highest hardness three times, while the ratio of 40 wt% CuO and 60 wt% TiO_2 reaches once. It seems that there is a tendency that when the total content of added nanoparticles is ranging from 5 to 15 wt%, the addition of both nanoparticles with similar amounts of CuO and TiO_2 shows the best hardness enhancement. This might be explained as follows: When both nanopartcicles added with

Table 2 Rockwell hardness test results

Group name	Group number						
	G1	G2	G3	G4	G5	G6	Average standard deviation
NCTx-1	81.7	80.8	79.3	83.8	84.6	85.3	3.8
NCTx-2	77.8	80.2	80.3	83.8	83.7	86.2	3.6
NCTx-3	81.6	78.9	84.3	85.2	86.4	83.4	4.2
NCTx-4	78.1	78.8	83.0	83.0	82.8	77.8	3.5
NCTx-5	79.6	81.1	85.9	84.3	84.8	85.9	3.6
NCTx-6	83.5	79.9	85.7	85.3	84.6	81.4	3.8
Average of each group	80.4	79.9	83.1	84.2	84.5	83.3	–
Control	79.9						4.0

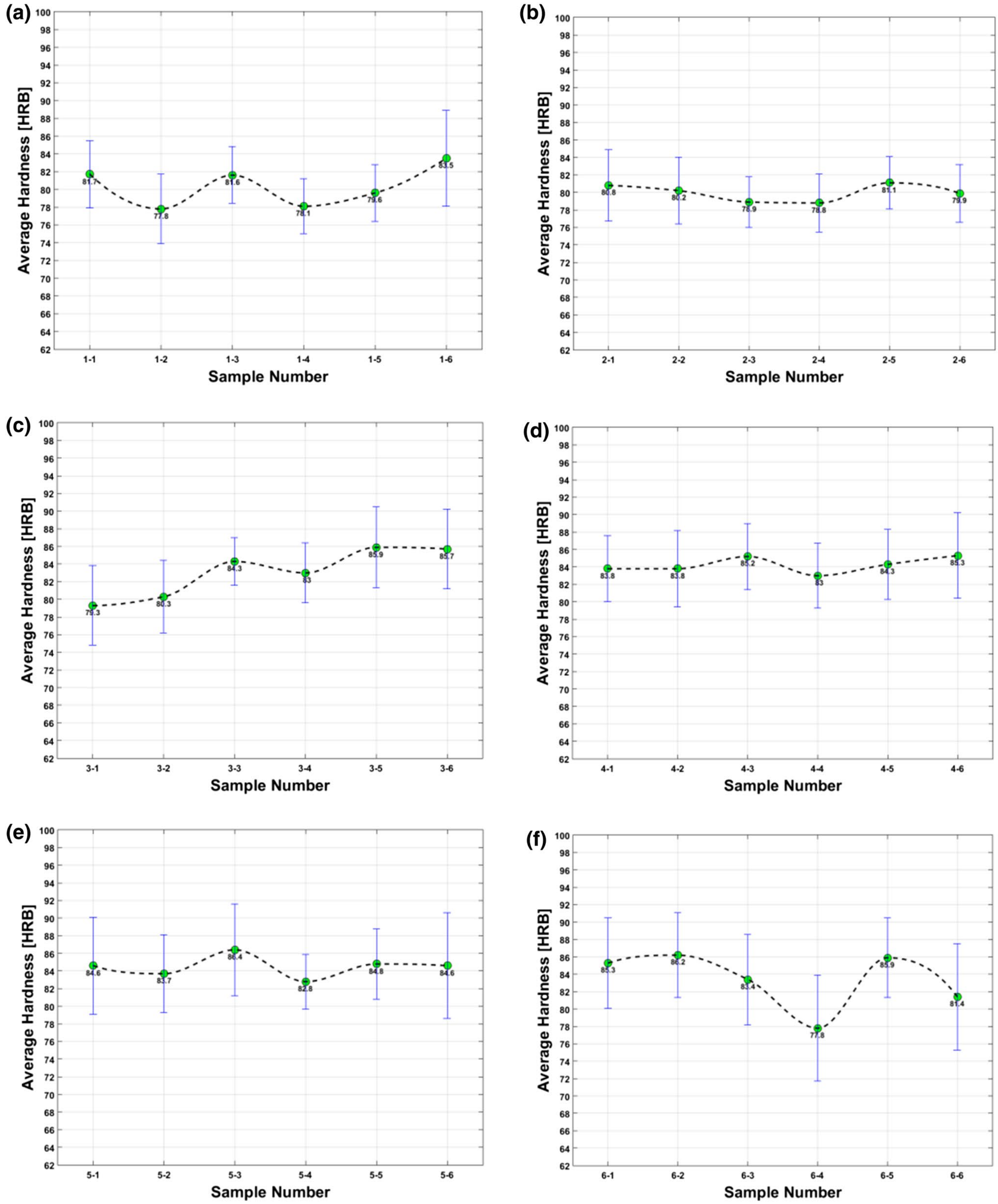

Fig. 6 Rockwell hardness for **a** group 1, **b** group 2, **c** group 3, **d** group 4, **e** group 5, and **f** group 6

similar amounts of CuO and TiO_2, the addition level of individual kind of nanoparticle would be far uder the 15 wt% limit of total level. Thus, voids in concrete can be filled with fillers (nanoparticles) of different sizes to achieve a finer and denser microstructure as shown in Fig. 5e for NCT5-3. While when the addition amound of single kind of nanoparticles is much more than the other, for instance, 12 wt% of CuO and 3 wt% of TiO_2, 12 wt% of CuO could be excess for filling relatively large voids, while 3 wt% of TiO_2 is insufficient to fill in all the relatively small voids. In this case, the hardness of a combined group with one kind of nanoparticles much more than the other will not be as good as those shown by groups with similar amounts of both kinds of nanoparticles.

Figure 7 shows the hardness of different subgroups, with each subgroup representing a specific ratio of added nanoparticles. In subgroup 1, there is a sudden drop of hardness from group 2 to 3. In subgroups 3 and 6, there is an abrupt decrease in hardness from group 1 to 2. Subgroup 5 encounters a sudden increment in hardness at group 3. These sudden changes break their hardness enhancement tendency, which recovers soon at the next subgroup. This uncertainty could be caused by the low amount of added nanoparticles in the first three groups, in which the amount of nanoparticles is not yet sufficient to fill all the voids, and there would have been a combination of improved fine grains and unmodified large grains. SEM observation (not shown) illustrates that group 1 samples (only 1 wt% nanoparticles added) have similar microstructures to the plain concrete sample. Group 5 and 6 samples (10 and 15 wt% nanoparticles added) show more uniform and finer grain structures, and groups 2 and 3 with medium percentages of nanoparticles added show partially modified structures. This incomplete size reduction of the grains for group 2 and 3 samples may enlarge the inhomogeneity in grain sizes and therefore result in more uncertainty in hardness measurements.

Figure 8 shows the hardness vs the percentage of added nanoparticles for the samples with either CuO or TiO_2 nanoparticles (subgroups 5 and 6).

As shown in Fig. 8a, samples with 5 wt% added nanoparticles show the highest hardness. For groups 2 and 6, the samples with TiO_2 nanoparticles show higher hardness than those with CuO nanoparticles, while for groups 1 and 4, CuO nanoparticles yield a higher hardness value. Samples in groups 3 and 5 show similar hardness. The average hardness values for TiO_2 and CuO nanoparticles are 83.6 HRB and 83.4 HRB, respectively, which are very close to each other. Therefore, there is no huge difference in hardness enhancement for the two kinds of nanoparticles. This is reasonable because these two kinds of nanoparticles modify the microstructure of concrete in a similar way: being fillers to decrease porosity and catalysts to enhance the hydration reaction.

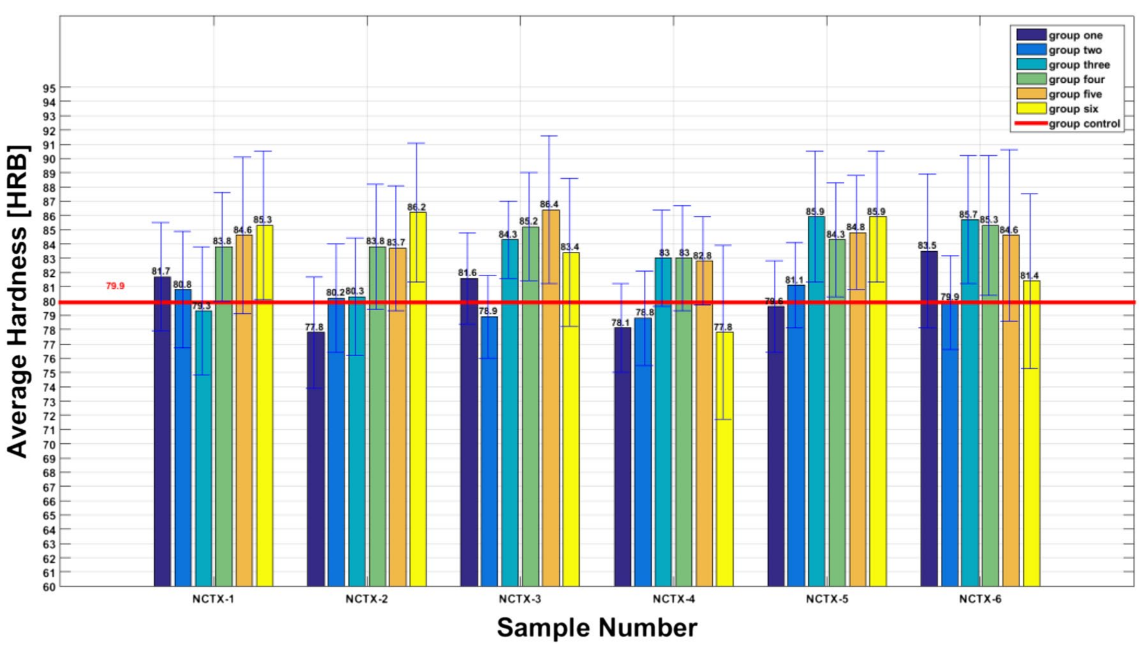

Fig. 7 Bar chart of Rockwell hardness of different subgroups with the same additive ratio

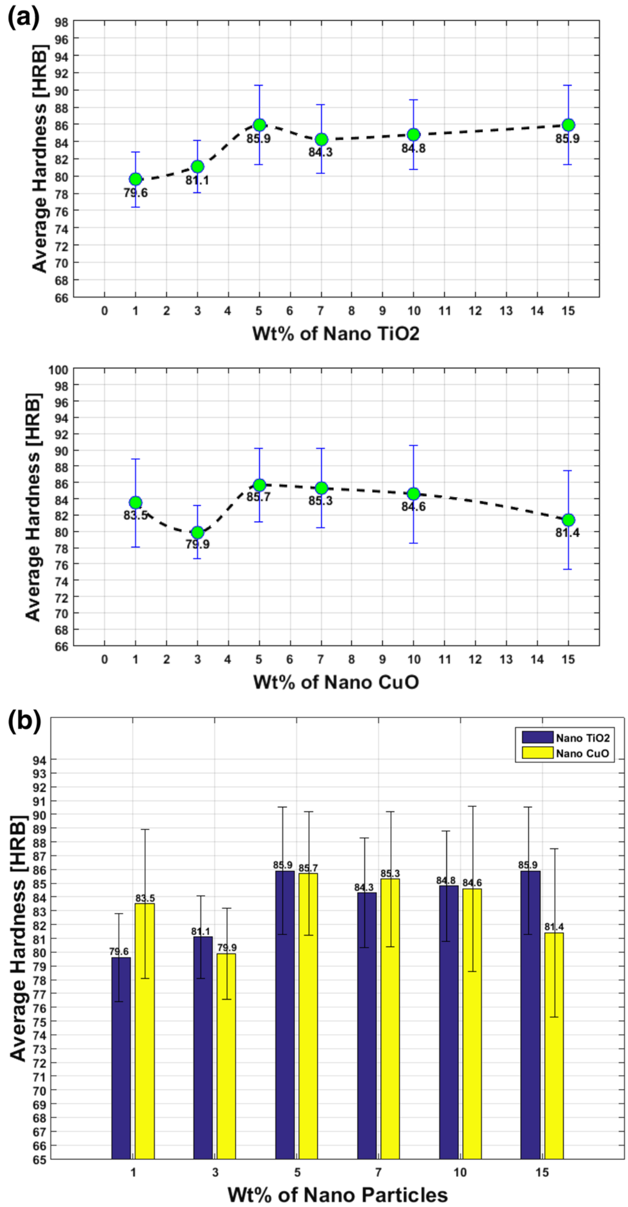

Fig. 8 **a** Line chart and **b** bar chart of Rockwell hardness of concrete with added TiO_2 or CuO nanoparticles

4 Summary and conclusions

The effect of adding CuO and/or TiO_2 nanoparticles on the hardness of cement mortar has been systematically investigated. The results have demonstrated that the addition of nanoparticles can refine the grains and densify the microstructure of the nanocomposites and thus increase the hardness. More detailed results are summarized as follows:

1. The addition of nanoparticles can effectively enhance the hardness of cement mortar. The enhancement is more obvious when the percentage of nanoparticles added is larger than 5 wt%. 4 wt% of CuO and 6 wt% of TiO_2 addition gives the highest hardness.
2. There is a tendency for the hardness to firstly increase and then decrease as the total amount of added nanoparticles increases. The increment could be due to the improvement in microstructure and hydration process by the addition of nanoparticles, while the reduction could be caused by excess nanoparticles and less cement which creates defects and weakens the formation of $Ca(OH)_2$ crystals.
3. The composite samples show the highest hardness enhancement when CuO and TiO_2 were added with close percentages as two kinds of nanoparticles, being of different sizes, would fill into voids of different dimensions in the concrete, thus resulting in a denser structure.
4. The effect of CuO or TiO_2 nanoparticles alone on enhancing hardness is similar due to their similar enhancement mechanisms.

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Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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