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ORIGINAL ARTICLE

Heat Transfer Characteristics of Metallic Body during Quenching in Saturated Nanofluids

H. Ismail[1], M. Z. Sulaiman[1,*] and T. Okawa[2]

[1]Faculty of Mechanical and Automotive Engineering Technology, Universiti Malaysia Pahang, 26600 Pekan, Pahang, Malaysia
Phone: +6094315407
[2]Department of Mechanical and Intelligent Systems, The University of Electro-communications, 1-5-1, Chofugaoka, Chofu-shi, Tokyo 182-8585, Japan

ABSTRACT – Heat transfer characteristics (HTCs) of a metallic rod during quenching were investigated in saturated water-based nanofluids and the results were compared to distilled water. In this study, a 50 mm length of cylindrical copper rod with a diameter of 15 mm was rapidly quenched at an initial temperature of 600 °C in saturated water-based nanofluids under atmospheric pressure. Three different types of nanoparticles (Al_2O_3, SiO_2 and TiO_2) were used to obtain water-based nanofluids with 0.001% particle volume fraction. In this experiment, heat transfer rates were evaluated using the cooling curves (temperature vs time) of the copper rod quenched in different quenching media. Results showed that the quenching heat transfer in nanofluids was stochastic during the first quenching, with SiO2 showing a deterioration in HTC, enhancement was observed in Al_2O_3 and TiO_2 compared to distilled water in the present work. However, after successive quenching, from second to seventh, all nanofluids demonstrated enhancement in the cooling time, with quenching in TiO_2 nanofluid showing the most significant enhancement compared to distilled water. Some deposition of nanoparticles on the surface of the rod was noticed, due to oxidation in distilled water quench, and mixed oxidation and nanoparticles deposition in the nanofluid quench. The present work suggested oxidation lead to deterioration of HTC, but a mixed effect of oxidation and nanoparticles deposition during the quenching process, altered the surface roughness of the rod surface and improved wettability. Hence, it was possible that the effect of surface structure vapour during film boiling influenced the dynamics of the bubbles in nucleate boiling and therefore enhanced the rapid cooling of the rod.

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INTRODUCTION

Quenching refers to the rapid cooling of a hot object by immersion into a cold fluid. It is an effective cooling technique in various engineering fields, such as heat treatment of steel and the safety procedures of nuclear power plants. During quenching, cooling curve shape analysis is used to evaluate quenchants heat transfer performance (QHTC) in several boiling regimes. The process is initially dominated by film boiling, where a continuous vapour layer completely separates the liquid from the solid surface. In this phase, the heat transfer occurs through conduction and radiation from the surface of the liquid through the vapour layer; therefore, the liquid takes a much longer duration to evaporate than at lower surface temperatures. However, as the temperature reaches the Leidenfrost point, liquid-solid contacts occur briefly and occasionally at discrete locations on the solid surface. If bubble nucleation occurs at these contact points, the vapour film is disrupted, and the heat transfer regime changes from film boiling to transition boiling. The vapour film gradually disappears, and the transition to nucleate boiling occurs, which is the ideal working condition for the quenching process since it produces the highest heat transfer rates. When the cooling curve shifts to the right, it indicates that the heat transfer performance is increased or vice versa and the steeper cooling curve represents an enhancement in the rate of heat transfer.

Primarily, the quenching process is applied in metallurgical industries to acquire specific material characteristics through the rapid cooling of a workpiece in water, oil, or air. On top of that, the application of quenching is also anticipated in a nuclear reactor to be used as an emergency cooling procedure, known generally as the emergency core cooling system (ECCS) [1]. The ECCS plays a significant role as a countermeasure in nuclear reactors during a loss of coolant accident (LOCA), where the coolant that is used as a countermeasure to control the temperature of the reactor vessel is rapidly lost, which could initiate an accident if a sudden increase in power occurs within the nuclear core. The fuel pins will become extremely hot in such cases. Under these conditions, the ECCS injects cold water and replenishes the reactor vessel, quenching the fuel rod in the nuclear power plant during a severe accident [2]–[4]. Therefore, ensuring optimum heat transfer rates during LOCA is critical. Without these safety systems, the core of the nuclear power plant could become damaged [5].

The conventional heat transfer fluids commonly used to remove the generated heat are water or water solutions, oils, molten-salt baths, polymer solutions (ethylene and glycol), fluidised beds, and compressed gases [6]. Compared to most

*CORRESPONDING AUTHOR | M. Z. Sulaiman | zuhairi@ump.edu.my

materials in general, these liquids have low heat transfer characteristics. Hence, new variations of fluids have been produced, known as nanofluids [7]. These nanofluids could be made by dispersing nanoparticles in a base fluid. Many experiments conducted on nanofluids have proven that adding a minuscule amount of nanoparticles to the base fluid has a remarkable effect on the thermophysical properties of the liquid. Consecutively, these enhancements to the thermophysical properties of the quench media could enhance the heat transfer rates during quenching [8]. Some frequent nanoparticles that were extensively used in recent studies were the particles of metal and metal oxides (Cu, Zn, CuO, Al_2O_3, TiO_2), carbon-based nanoparticles (MWCNTs) and polymeric nanoparticles [2], [9]–[19]. By dispersing the nanoparticles in a quench media, the quenching process of steel occurs at faster rates, with enhanced cooling capabilities, more significant impact strength and lower steel dimensional modifications than conventional fluids [20].

In recent years, extensive studies have been performed on the effect of nanoparticle addition on the quenching behaviour of the metal. Since conventional fluids could not achieve the desired heat transfer rate, it is anticipated that nanofluids will accelerate this transition. However, based on recent literature, certain inconsistencies were found in the quenching heat transfer coefficients (QHTC) after using nanofluids, which were reported to have increased, decreased or been unchanged. In 2004, Park et al. [21] studied a stainless steel sphere's quenching in different concentrations of Al_2O_3/water at different subcooled conditions. It was found that the HTCs when using nanofluids were more extensive as the subcooled conditions decreased. The authors also noticed that the effect of nanoparticle concentrations of higher than 5 vol% is negligible during quenching. Kim et al. [1] conducted research on the quenching of a small stainless steel sphere in Al_2O_3/water nanofluids at much lower concentrations (0.001, 0.01 and 0.1 vol%), quenched in saturated conditions. The authors discovered that some nanoparticles were deposited on the sphere surface during the quenching process and also after multiple re-quenching tests using the same sphere. In their experiment for alumina, it is suggested that no concentration effects are made in the case of the first run. However, for the repetitive case, higher concentration showed improvement of the cooling curve. It was also noticed that the nanoparticles deposited on the surface of the specimen accelerated the quenching process in that the transition from film boiling to nucleate boiling occurred at greater temperatures compared to a new and clean surface.

However, the study by Lotfi and Shafii [22] who quenched using a silver sphere in subcooled Ag and TiO_2 water-based nanofluids revealed that the trend for the cooling curves of the heated sphere during the first quenching process in nanofluids was indistinguishable from the trend when pure water was used. It was also observed that the result of subsequent tests on the unwashed sphere was more vigorous with the existence of nanoparticles that were deposited on the sphere surface. It was assumed that the nanoparticles deposited on the sphere surface acted as an insulator for the sphere and decreased its surface temperature, given that the TiO_2 layer formed having a larger thermal resistance.

Meanwhile, Vignesh and Narayan [23] studied the cooling performance of an alloy rod during quenching in distilled water and MWCNTs/water nanofluid. It was noticed that the rate for the heat transfer was reduced when the rod was quenched with 0.01 and 1.0 g/L of nanoparticles. The reduced efficiency may be due to the relative effects of nanoparticle deposition on the surface of the rod and its Brownian motion. This deposition acts as a barrier to heat transfer and decreases the rate of heat transfer. Dasgupta et al. [24] conducted an experiment to determine the performance of an SS304 rod during quenching in Al_2O_3/water nanofluids at saturated and subcooled conditions. It was noticed that the quenching times for water and nanofluids were similar during subcooled conditions. However, the heat flux behaviour during quenching in nanofluids differs from quenching in pure water. The initial film boiling phase in nanofluids is faster, but the maximum heat flux for water is greater, and it decreases as nanofluid concentration increases. Irregularities in the heat flux during the nucleate boiling region were also noticed, which could be caused by the deposition of nanoparticles on the rod surface.

Recent literature stated that the likely causes of the inconsistencies were the types and concentrations of nanoparticles being used. In many cases, the usage of Al_2O_3 nanoparticles shows a significant increase in QHTC compared to the use of SiO_2 nanoparticles. Hence, the present study evaluated the cooling curve of a high-temperature copper rod quenched, representing the QHTC performance in various nanofluids (Al_2O_3, TiO_2 and SiO_2) in relatively low concentration. In this paper, the effects of the nanoparticle depositions on the rod surface and oxidation conditions during quenching were explored and compared to the heat transfer characteristics when distilled water was used.

METHODS AND MATERIALS

Nanofluid Preparation

Three types of nanoparticles were used in this research; (1) Aeroxide Alu C (Aluminium Oxide, Al_2O_3) particles with d_p of 13 nm, (2) Aerosil 90 (Silicon Dioxide, SiO_2) particles with d_p of 20 nm and (3) Aeroxide TiO_2 P25 (TiO_2 particles with a mixture of anatase (80%) and rutile (20%) crystal structures with an average d_p of 21 nm). These were obtained from Aerosil Corporation in powder form. The properties of the nanoparticles used are given in Table 1.

Table 1. Properties of nanofluids used [25].

Nanoparticles	Size (nm)	Density (kg/m^3)
Al_2O_3	13	3880
SiO_2	20	2200
TiO_2	21	4175

The nanofluids were prepared using a two-step method and by mixing the nanoparticles directly with distilled water. Using this method, the nanoparticles were measured and weighted in primary dilution of 75 ml in a test tube. The measured weight was then converted into the volume of the nanoparticles and then successively suspended in the designated volume of the base fluid to achieve a concentration of C= 0.001 vol%. Prior to the final dilution process, the suspension was then stirred for 15 minutes using a magnetic stirrer to achieve homogeneity and was then sonicated in an ultrasonic bath (Bransonic® CPXH Ultrasonic Bath) for 1 hour just before the experiments were conducted, based on our previous work [26]. During the preparation process, no dispersant or surfactant was added to avoid altering the fluid's chemical and thermo-physical properties.

Experimental Setup

Figure 1 shows the schematic drawing of the copper rod (50×15 mm diameter), and the thermophysical properties of the test specimen are displayed in Table 2. Figure 2 shows the test rig used in conducting the quenching experiments. The main components used in this study were a borosilicate quench pool (diameter 130×180 mm), an electric melting furnace, a hotplate, and a data acquisition system. K–type thermocouples were used to obtain the temperature measurements of the centre of the rod and the quench media. The uncertainty in temperature measurements was ±1.5 °C. The National Instruments data acquisition (NiDAQ) system was used to record the temperature-time data during quenching.

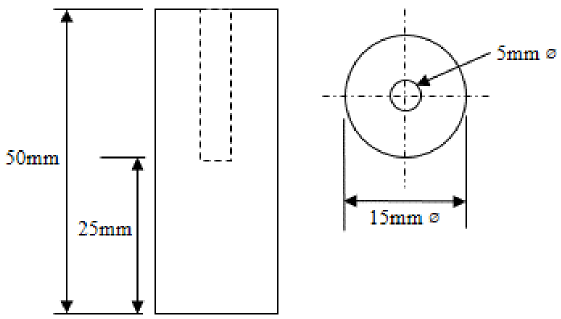

Figure 1. Schematic drawing of copper rod.

Table 2. Thermo-physical properties of the test specimen.

Properties	
Material	50×15 mm ø copper cylinder
Density, ρ	8960 kg/m^3
Specific heat capacity, c_p	376.812 J/kg·K
Thermal conductivity, K	385 W/m·K

Distilled water inside the quench pool was first heated to a saturated condition of T_{sat}= 100 °C using a hotplate, and the prepared Al_2O_3 nanofluids were then injected into the quench pool to achieve a concentration of C = 0.001 vol%. The temperature of the quench pool was maintained at saturated conditions throughout the experiment. At the same time, the copper rod was heated to 620 °C using an electrical furnace. The copper rod was then removed from the furnace and left suspended above the quench pool until the temperature of the centre of the rod reached 600 °C before it was quickly immersed in the quench pool. Once the specimen and quench media were at thermal equilibrium (~100 °C), the experiment was ended. The temperature data obtained from the quenching process was collected and analysed. Then, after the first quench, the rod was subsequently quenched again for another six times with the interval period of approximately 45 minutes, with the same rod surface but without any changes to the quench media during each test, to investigate the surface effect on the rate of heat transfer. Before starting the following test in SiO_2 and TiO_2 nanofluids, the surface of the rod was polished using 1500 grit sandpaper and PIKAL polishing paste to remove any residue from the previous tests. The surface was then wiped clean using acetone. In these experiments, cooling curves were drawn to represent the quenching heat transfer performance in various conditions. A centre temperature point at T= 250 °C, where the fluid is in the nucleate boiling region, was selected and analysed.

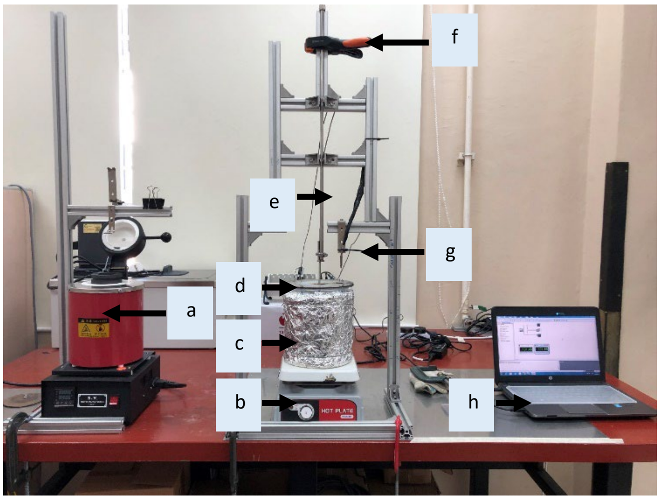

Figure 2. Quenching experimental setup where: (a) S.Y. Electric melting furnace, (b) Favorit Hotplate, (c) quench pool insulated with glass wool, (d) polycarbonate cover, (e) 5 mm K-type thermocouple to read copper temperature, (f) clamp as a rod holder, (g) 1 mm K-type thermocouple to verify water temperature, (h) laptop connected to NiDAQ system.

RESULTS AND DISCUSSION

Figure 3 shows the preliminary quenching test using base media, which is distilled water. The specimen was quenched at least three times repetitively using a fresh, clean rod. The quenching results demonstrated an approximately similar cooling curve that almost converged to a single one, indicating the reliable repeatability of the present work. As depicted in Figure 3, during test 1 and test 2, the copper rod took 43.5 s to reach T=250 °C in the boiling region, while in test 3, it took 45 seconds, a delay of only 1.5 s. The results indicate the excellent repeatability of the quenching experiments. Hence, the results obtained using distilled water as the base media were used as a reference for the performance of quenching heat transfer characteristics and later will be compared to different types of nanofluid-based media.

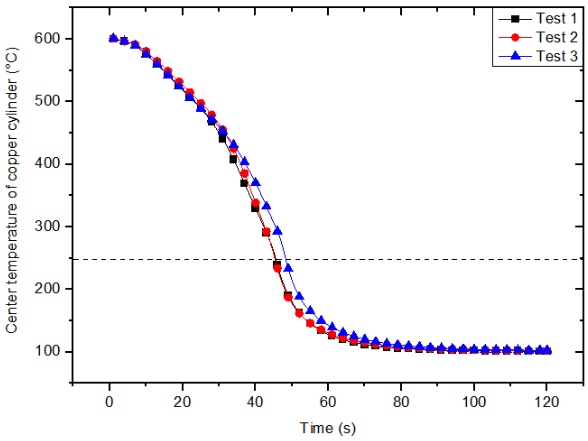

Figure 3. Repeatability test for quenching of copper rod in distilled water.

Figure 4 shows the quenching curves, which represent the repeated quenching of a copper rod in distilled water. It can be seen that the first quench using a clean rod had a faster heat transfer rate, as the time taken for the rod to reach 250 °C was 43.5 s. The subsequent tests using an unwashed rod in the same quench media showed a decrease in heat transfer, with a much longer time taken for the rod to cool. Based on the figures, it could be seen that the cooling curve drastically shifted to the right during the second quench, as the rod took 65.5 s to reach 250 °C. However, the cooling curves gradually shifted to the left during the third quench with a much higher magnitude after the fourth quench, as seen in Figure 4.

As shown in Figure 5(b), the surface of the rod changes from shiny to dull after the first quench. Some visible dark spots and oxidation occurred on the surface after quenching in distilled water. In Figure 5(c), 5(d) and 5(e), it can be seen that the deposits on the rod gradually increased, which at the same time reduced the time taken for the rod to reach 250 °C in the boiling region, from 65.5s during the second quench to 63.5s in the fourth quench. Figure 5(e) shows that a higher deposition intensity occurred on the rod surface, resulting in a shorter time for the rod to cool during the fifth quench, which occurred at 56.5 s of quenching. However, subsequent tests showed only a slight difference in the surface structure of the rod, as shown in Figures 5(f) and 5(g). A similar result could be seen in a study by Kim et al. [27], which mentioned that the dark spots were not due to emissivity heterogeneities on the rod's surface since the dark spots moved on the surface and afterwards disappeared. No dark spots were observed throughout the first or repeated cooling cycles in the air. Thus, it could be said that the dark spots seen in Figure 5 are associated with water/surface interaction. From the results obtained, the change from a shiny to a dull surface during the first quench had highly affected the rate of heat transfer of the copper rod. The heat transfer performance deteriorates when a dull surface is used during quenching. However, as the number of quenches increases, more depositions occur on the surface of the rod, which increases the quenching heat transfer performance of the copper rod in the present experimental work. The study by Hwasung et al. [28] also reported the sensitivity of the surface oxidation due to the quenching in water, and an example of the oxidation was qualitatively discussed in the study of Lee et al. [2]. The oxidation could lead to a different vapour film collapse mode, as studied by Lee et al. [29]. Thus, it is most probable that in the present work, the deterioration of HTC could be associated with the thin layer of oxidation formed and started to insulate the quenched surface.

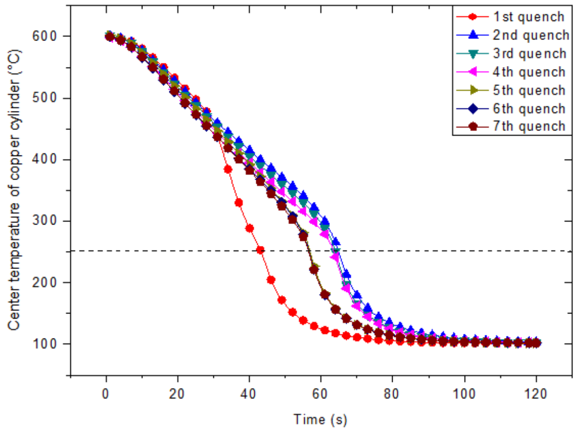

Figure 4. The cooling curves of copper rod in distilled water.

(a) (b) (c) (d) (e) (f) (g) (h)

Figure 5. The surface structure of copper rod during quenching in distilled water; (a) after polishing, (b) 1st quench, (c) 2nd quench, (d) 3rd quench, (e) 4th quench, (f) 5th quench, (g) 6th quench and (h) 7th quench.

When quenching in Al_2O_3 nanofluid, the first quench showed a significant enhancement in the cooling time compared to using distilled water. The rod took only took 38.5 s to reach 250 °C in the boiling region, while during the second quench using an unwashed rod, the performance deteriorated rapidly, as the duration for the rod to cool increased to 58.5 s. However, the third quench improved heat transfer rates, as the rod took a shorter time to cool and the cooling curves gradually shifted to the left after each subsequent test, as shown in Figure 6. During the seventh quench, the rod took only 43.5 s to reach the boiling region.

The surface structure of the rod was also observed after each test in Al_2O_3 nanofluid. Based on Figure 7, there was a noticeable increment in the number of nanoparticles deposited on the surface of the rod. Like the results for distilled water, the surface appearance became dull after the first quench, as shown in Figure 7(b). After each subsequent quench, the nanoparticles deposited on the surface of the rod were also increased. It was concluded that the increase of surface roughness due to the deposition of nanoparticles was responsible for the improved quench boiling heat transfer [17]. In this case, the nanoparticles deposited showed a positive increment in the quenching of the copper rod, similar to the results obtained in the study by Kim et al. [27].

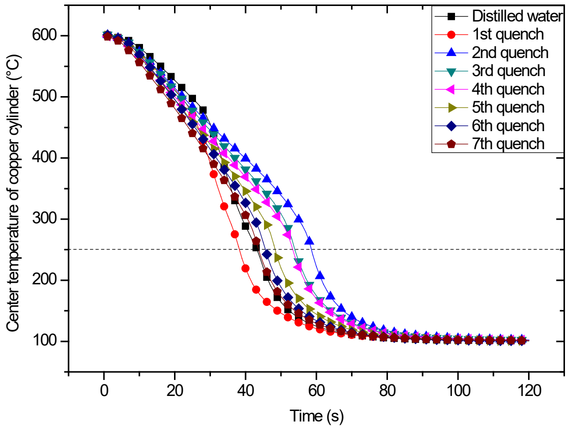

Figure 6. The cooling curves of copper rod in Al_2O_3 nanofluid in 0.001 vol% after repeated runs.

(a) (b) (c) (d) (e) (f) (g) (h)

Figure 7. The surface structure of copper rod during quenching in Al_2O_3 nf at 0.001 vol% (a) after polishing, (b) 1st quench, (c) 2nd quench, (d) 3rd quench, (e) 4th quench, (f) 5th quench, (g) 6th quench and (h) 7th quench.

The graph in Figure 8 shows that the first quench of the clean rod in SiO_2 nanofluid took a much longer time to cool, 53.5 s, compared to using distilled water, which is a 22% reduction in cooling time. However, the cooling rate worsened in the second quench using an unwashed rod, as the rod took 56.5s to reach 250 °C. Similarly, the subsequent tests showed a significant enhancement in the cooling time of the rods compared to the second quench. The seventh quench using the same rod ended the film boiling region at 44 s, a much faster duration than quenching for the fresh, clean rod. Based on Figure 9(h), the SiO_2 nanoparticles are visible after repetitive quenching. Resembling the study by Kim et al. [30], the quenching heat transfer performance was enhanced only after repetitive quenching due to the nanoparticles being deposited on the surface of the rod, which increases its surface roughness and could disrupt the formation of bubbles during film boiling.

During quenching in TiO_2 nanofluid, the cooling time for the copper rod was much faster than quenching using distilled water, and the TiO_2 nanofluid showed a 9 % enhancement as it reached the rod temperature of T=250 °C with 39.5 s of quenching, as shown in Figure 10. During the second quench using an unwashed rod, the time taken to cool the rod increased to 51.5 s because of the difference in the surface roughness of the rod. Figure 11(b) shows the surface of the rod after the first quench. Afterwards, the cooling time during quenching of the unwashed rod gradually shortened, and it had reduced to only 41.5 s after the seventh quench, which is almost identical to quenching in distilled water. From this figure, it can be seen that repetitive quenching of an unwashed rod could increase the quenching performance of copper rods when using TiO_2 nanoparticles. Lotfi and Shafii [22] reported that the nanoparticle deposits that occurred on the surface acted as a thermal insulator and reduced the temperature of the outer surface of the rod due to the higher thermal resistance of the TiO_2 coating. In addition, the higher emissivity of the TiO_2 layer led to an increase in heat transfer and the faster cooling of the outer surface of the specimen. These prevented the formation of a stable vapour film around the rod, which subsequently facilitated the rapid quenching through the mode of nucleate boiling, bypassing the film boiling mode [22].

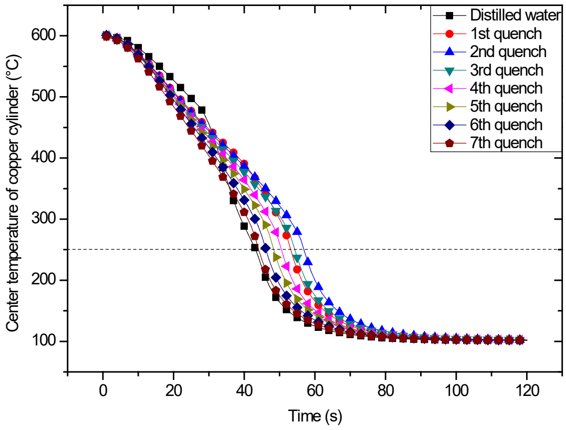

Figure 8. The cooling curves of copper rod in SiO_2 nanofluid at 0.001 vol% after repeated runs.

(a) (b) (c) (d) (e) (f) (g) (h)

Figure 9. The surface structure of copper rod during quenching in SiO2 nf at 0.001 vol% (a) after polishing, (b) 1st quench, (c) 2nd quench, (d) 3rd quench, (e) 4th quench, (f) 5th quench, (g) 6th quench and (h) 7th quench.

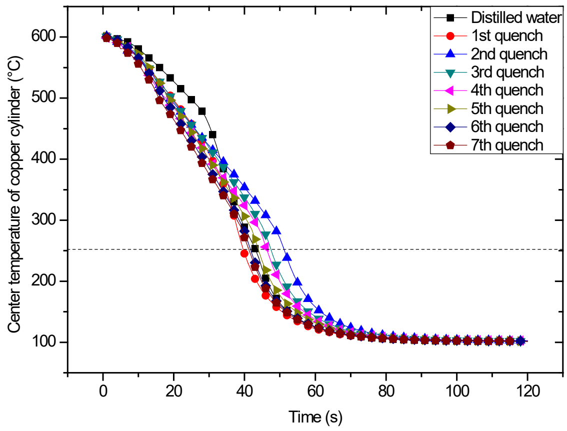

Figure 10. The cooling curves of copper rod in TiO_2 nanofluid at 0.001 vol% after repeated runs.

(a) (b) (c) (d) (e) (f) (g) (h)

Figure 11. The surface structure of copper rod during quenching in TiO_2 nf at 0.001 vol% (a) after polishing, (b) 1st quench, (c) 2nd quench, (d) 3rd quench, (e) 4th quench, (f) 5th quench, (g) 6th quench and (h) 7th quench.

Figure 12 shows the comparison of the time taken for the rod to cool after multiple quenching in different types of nanofluid and distilled water. The figure shows similarities in the trends of each quench using the three types of nanofluid and distilled water. However, after the second quench, the trend for nanofluids showed an enhancement compared to that of distilled water, as it took less time for the copper rod to reach the temperature of 250 °C in the boiling region. After the seventh quench, TiO_2 nanofluids showed the most significant enhancement, which was 26.5% faster than using distilled water. Based on the literature, enhanced cooling performance is attributed to the high wettability of a thin layer that forms on the surface by deposition of nanoparticles which prevents a stable vapour film from forming around the rod [31].

Based on the results obtained, the surface roughness of the rod has a significant effect during the quenching process, either in distilled water or nanofluids. Hence, it is appropriate to highlight the impact of surface morphology with the use of photographic observations, as shown in Figure 5(h), Figure 7(h), Figure 9(h) and Figure 11(h). However, it is still unclear whether the nanoparticles with a low concentration in distilled water could enhance the vapour generation of the copper rod to further increase the heat transfer rates. It is possible that mixed oxidation and nanoparticles deposition with each other causing stochastic cooling performance. It should be noted that the oxidation may form a thin layer, as described in studies by Mimura et al. [32], that leads to a reduction in nucleation spots. It is also recommended in the future that the changes in the surface structure of the rod are measured, and the heat transfer rate during quenching are investigated more extensively.

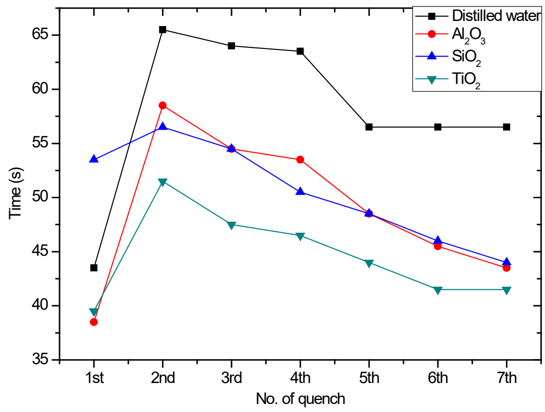

Figure 12. Comparison of the time taken for the copper rod to reach the temperature of 250°C in the nucleate boiling region after each subsequent quench for different types of quench media at *C*= 0.001 vol%.

CONCLUSION

In this study, quenching experiments on a heated copper rod in different types of nanofluid at a low concentration of 0.001 vol% were conducted to study the quenching heat transfer compared to distilled water. A rod with a diameter of 15mm and a length of 50 mm, at an initial temperature of 600 °C, was tested in saturated conditions. The following results were obtained:

i. Based on the cooling time for nanofluids, the heat transfer performance during quenching in a low concentration of nanofluids showed a dramatic enhancement in Al_2O_3 and TiO_2 compared to distilled water with 13.3 % and 12.6 % increment in the cooling time, respectively, except for SiO_2 with 22 % of decrement in cooling time during the first quenching.
ii. In repetitive quenching, the HTC showed reduction as the cooling time took slightly longer in repetitive quenching than the initial quenching for all nanofluid-based liquids, indicating that the HTC deteriorates for the second quenching distilled water and all nanofluid based fluids. However, relative enhancement in cooling time comparison between distilled and nanofluid based shows up to 40 % to 63 % reduction in cooling time in seven series of repetitive cooling. The TiO_2 nanofluids exhibited the highest enhancement in cooling time and almost similar results for Al_2O_3 and SiO_2 in the present concentration.
iii. The quenching of a clean rod in the first quench resulted in better HTC compared to subsequent quench for all fluids. Oxidation in repetitive quenching is strongly believed to have reduced the quenching heat transfer. However, after repetitive quenching until the seventh quench, the build-up of a nanoparticle deposition layer revealed a significant increment in the quenching performance of the nanofluid. The nanoparticle deposition has a positive effect on the quenching of the rod.

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