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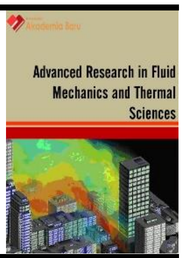

Thermal Conductivity and Stability Studies of Cooking and Waste Cooking Oil as a Based Fluid of TiO_2 Nanofluid for Carbon Steel Quenching Process

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ABSTRACT

Selection of quench media is important as it depends on the hardenability of the metal alloys, component's thickness and geometry. Recently, oil and water are frequently used as a quench media in heat treatment industries. Improper quench media will cause the material to become brittle, suffers from geometric distortion, or having a high undesirable residual stress in the components. Oil was used as the fluid base in this research as the main observation focus. To obtain TiO_2 nanofluids and explore thermal conductivity and stability of TiO_2 nanofluids, the two-step method was introduced to prepare the nanofluids with 4 types of different cooking and waste cooking oil as a base fluid (Palm oil, sunflower oil, canola oil and corn oil). TiO_2 powder will be mixed with oil fluid by using magnetic stirrer for 1 hour and ultrasonic bathing. Observation method used in 30 days with thermal conductivity and zeta potential to evaluate stability of each nanofluid specimens. Heat treatment onto carbon steel applied and the sample were heated at 950°C for 90 minutes and then quenched in difference oils. Microstructure and hardness analysis applied to get the result. It can conclude, adding nanoparticles in oil base fluid will enhance thermal conductivity and improve thermal performance of heat transfer of the base fluid. Quenching process with oil-based nanofluid will produce martensite structure and waste sunflower and corn oil with TiO_2 it's not stable after 30 days observation but the palm and canola oil with TiO_2 can achieve the highest hardness test and thermal conductivity.

1. Introduction

Thermal oil is one of the thermal fluids that widely used in heat transfer applications. Most of engineering applications (e.g.; automotive, aerospace, marine, military industries, internal combustion engines, compressor, gearboxes and processing equipment) where high temperature are the main obstacles need the used of thermal oils [1-3]. Moreover, in machining, cutting fluids which

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is also heat exchangers play an important role in decreasing internal friction during metalworking operation.

In automotive industries, heat treatment is one of the important process to produce certain automotive parts. AISI 1045 carbon steel is one type of carbon steel that is used for pin spring or a body part of vehicle, and it must have high strength, toughness and excellent wear resistant. In order to have those mechanical properties, heat treatment can be used to design the materials with the desired properties. Heat treatment can be defined as a combination of two procedures, heating and cooling metal or alloy in solid state which arranged in time intervals in order to alter their physical and mechanical properties to meet desired engineering applications [4]. The process is started by annealing at higher critical temperature, hold for a certain time, followed by a rapid cooling process or known as quenching. Several quenchants are available in the market that have different cooling characteristics such as; water, brine solution, polymers, oils, salts and gases. For different components that has a variation in section thickness, a slow cooling rate of quenchant is preferable to reduce residual stresses. High rapid cooling quenchant is possible to induce cracking, especially on the critical part of the samples. To get more evenly distributed cooling and preventing from cracking, medium cooling rate quenchant such as oil can be used as an alternative, thus a better hardness depth and evenly distributed on the component. Recent researcher and modern technologies provide new methods and produce new quenchant that having various level of severities of quenching. Slower cooling rate of quenchant are more preferable, however produce lower hardness.

Several researches have been focused on the synthesizing of fluids that contains small amount dispersed nanoparticles (< 100 nm) which is known as nanofluid [5]. It is expected to give better thermal conductivity and lower specific heats than conventional fluids. The type of nanoparticles being studied or used can be categorised as some advanced structural materials with highest thermal conductivity such as graphene, CNTs, diamond etc [6]. High thermal conductivity such Au, Ag, Cu, Al, Fe, etc for some metallic simples and some metal or non-metallic compounds such as CuO, Al_2O_3, TiO_2, ZnO, Fe_3O_4, SiC, SiO_2 etc [7-16]. Titanium dioxide (TiO_2) nanoparticles are one of the nanoparticles commonly used to be a nanoparticle in the suspension because of it is chemically and physically stable. It also has low cost and commercial availability, as well as free from health hazards.

Kim *et al.,* [17] is one of the researchers who carried out a quenching process of steel and zircaloy spheres in several types of pure water based nanofluid (0.1% Al_2O_3, SiO_2 and diamond). Researchers also examined quenching process around a silver sphere at high temperature with deionized water based nanofluid of Ag and TiO_2. They found that the nanofluids deteriorated the quenching performance and boiling heat transfer coefficient. However, they observed that the pure water based nanofluid of Ag and TiO_2 prevented the film boiling and nucleate boiling time increased. Hence, the sphere was cooled faster which was due to the deposited particles on the surface [18]. Most of the research was focused on the water based nanofluids. However, water is one of the fastest cooling rate quenchant mediums that could lead to cracking of the component.

The present study is aimed to observe the effect of cooling rate using cooking oil based TiO_2 nanofluid on the microstructure and hardness property of the mild carbon steel. Besides, the thermal and stability of the cooking oil based TiO_2 nanofluid is investigated and compared to the waste cooking oil based TiO_2 nanofluid. The reason of using waste cooking oil due to social and economic development that leads to the more presence of restaurants, directly increase the waste cooking oil output [19]. This unreasonable waste cooking oil disposal may result in environmental pollution and reusing waste cooking oil for is unhealthy for human body [19]. In the field of nanofluid technology, it can be seen that not much researchers had used waste cooking oil as a base fluid of nanofluid [20,21]. Thus, this study is aimed to add more variety on the availability of the nanofluid that can be used for quenching process.

2. Methodology

This paper tells about materials, equipment and preparation methods will be further discussed during the experimental investigation for the quenching behaviour on mechanical properties (hardness) and microstructure of quenched samples. The thermal conductivity and stability of the quenching medium i.e. nanofluid also be investigated in this experiment.

Firstly, titanium dioxide (TiO_2) powder purchased from Sigma Aldrich (Specification of TiO_2 is summarized in Table 1) will be used for synthesizing TiO_2 nanofluid with different cooking and waste cooking oil as a base fluid (Palm oil, Sunflower oil, Canola oil and Corn oil). Stability test will be carried out for all specimens' oil to obtain the specimen with highest stability. Zeta potential measurement is used for stability evaluation in this study.

The material used in this study is high carbon steel and heat treatment processes on specimens were carried out. The all specimens' heat treated in a Nabertherm furnace at temperature of 950°C and then was hold for 20 minutes at same temperature, followed by oil quenching with 12 types of oil specimens. The details about treatment process, microstructure analysis and hardness test of each specimen are stated in this paper.

Titanium dioxide powder and 50 g oil fluid are weighed by using precision weight balance according to the calculated weight fraction between TiO_2 (Table 2). Firstly, weight of TiO_2 in 0.1% weight fraction between TiO_2 and 50ml oil fluid is calculated by using Eq. (1). Then from the weight of TiO_2 obtained, a weight fraction between TiO_2 is calculated.

$$\varphi = \frac{(W/\rho)_{\mathrm{TiO2}}}{(W/\rho)_{\mathrm{TiO2}} + (W/\rho)_{\mathrm{oil}}} \tag{1}$$

where ϕ is the volume fraction, W is weight and ρ is density.

Table 1
Properties of TiO_2 nanoparticle

Nanoparticles	Density (g/cm³)	Thermal Conductivity (m. K)$^{-1}$
Titanium dioxide, TiO_2	4.23	8.4

Table 2
Weight of TiO_2 with differences oil

Oil types	Weight Fraction (TiO_2)	Weight of TiO_2 (g)	Weight of oil fluid (g)
Palm oil Canola oil Sunflower oil Corn oil	0.1%	0.21	50

2.1 Stability Evaluation

Sedimentation technique that we apply by observing the substance of combination TiO_2 and difference oil. It can be said that when the TiO_2 powder sediment rapidly it can be concluded that the stability is lower. Same goes when the TiO_2 powder sediment slowly or take time to sediment it can say that the stability of the substance or nanofluid is higher.

In this experiment, to test the stability of the nanofluid with different cooking and waste cooking oil as a base fluid, 8 specimens of oil have prepared as shown in Figure 1. For reference sample, as-received cooking oils (palm, canola, corn and sunflower) are used. Figure 2 shows flow chart of the experiment with difference oils.

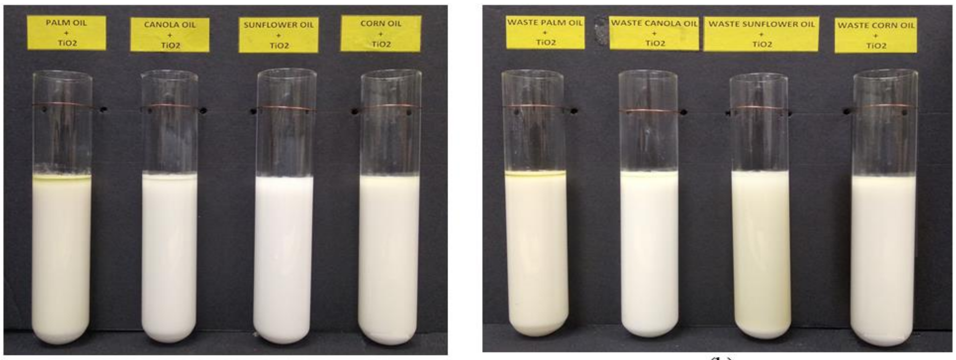


Fig. 1. Experiment setup of samples; (a) New cooking oil (b) Waste cooking oil

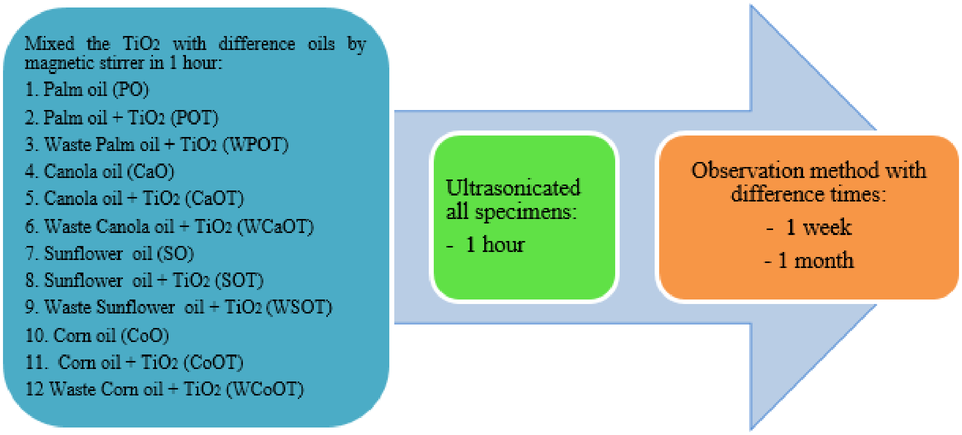


Fig. 2. Process flow of experiment

2.2 Zeta Potential Analysis

Besides centrifugation machine, zeta potential measurement is also used to evaluate stability of nanofluid specimens. Zeta potential is determined through the rate of a charged particles move in response to an electric field.

2.3 Thermal Conductivity Analysis

Thermal conductivity evaluation will be performed on the nanofluids which has highest stability after the second stability test. Thermal property analyser (TPA) will be used to evaluate the thermal conductivity of nanofluids. Thermal property analyser measures the average temperature rise of double spiral in probe to evaluate thermal conductivity of specimen.

2.4 Heat Treatment

The prepared specimens of high carbon steels will be exposed to the heat treatment processes as quenching. Each of the cooking oil will be divided into 3 samples as shown in Table 3 below.

Table 3
Type of oil base fluid

Description	Type of Oil (Sample)			
	Palm Oil	Canola Oil	Sunflower Oil	Corn Oil
Pure oil	PO	CaO	SO	CoO
New oil with TiO_2	POT	CaOT	SOT	CoT
Waste oil with TiO_2	WPOT	WCaOT	WSOT	WCoT

These specimens of high carbon steels were heated to the dual phase region (ferrite-martensite) at the temperature of 950°C for 90 minutes in furnace machine. Then, it was hold for 20 minutes at same temperature in the furnace and quenched in each specimen's oil quickly.

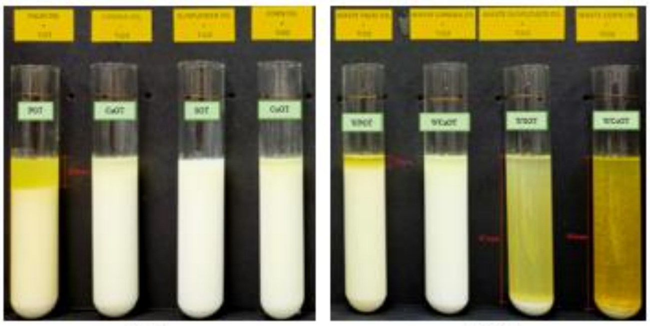
2.5 Hardness Test

The mechanical testing of the specimen was conducted by using hardness test. This Vickers hardness was used to measure the hardness of a specimen by calculating the size of an impression produced under load applied by a pyramid-shaped diamond indenter. The indenter employed in the Vickers test is a square-based pyramid whose opposite sides meet at the apex at an angle of 136°.

Firstly, the diamond was pressed into the surface of the specimen at load 49.04N and then the size of the impression (usually not more than 0.5 mm) was measured with the aid of a calibrated microscope. Next, the value of HRC was collected from the Digital LCD screen. There are 3 points of pyramid-shaped diamond was indented on the specimen to get the average value of HRC. Thus, one of the samples from each group was undergo hardness testing.

3. Results
3.1 Stability Analysis

TiO_2 nanofluid with different cooking and waste cooking oil as a base fluid that have been mixed using a magnetic stirrer for an hour. Then, the prepared sample were kept them standing and observed for 30 days as shown in Figure 3.



(a) (b)

Fig. 3. (a) New cooking oil (b) Waste cooking oil

Based on the experiment result, it found that SOT nanofluid is more stable than other cooking oil based TiO_2 nanofluid after 30 days observation. Figure 3(a) shown that SOT nanofluid is cloudier. For CaOT and CoOT has a slight sediment but not much. Only POT show a more significant of sediments as a layer between the occurrences of two parts oil and TiO_2. It can be concluded that POT is less stable due to TiO_2 density being unbalanced when mixed with palm oil.

Observation on waste cooking oil, the experiment result show that WCaOT nanofluid is more stable than other cooking oil after 30 days. For WPOT, showed a slight improvement in its stability than before as shown in figure but the WSOT and WCoOT showed the lowest stability. It can be concluded that the mixture of foreign substances present in the oil has a big effect on the density of TiO_2 and thus weakens its stability.

3.2 Zeta Potential Analysis

Zeta potential measurement is a second method to evaluate stability of nanofluid specimens. In this investigation, the thermal conductivity of cooking oil based on nanofluids was measured by zeta potential method, but this sample exhibited unexpected results after 3 measurements were made. This measurement will be repeated by performing the sonication process for more than 1 hour. Table 4 shown the result of all zeta potential measurement for each sample on cooking and waste cooking oil with nanofluid.

Table 4
Zeta Potential taken for nanofluid with different cooking oil

Oil Type	Sample Labelling	Zeta Potential (mV)
Palm Oil- TiO_2	POT	13.8
Waste Palm Oil- TiO_2	WPOT	22.1
Canola Oil- TiO_2	CaOT	27.8
Waste Canola Oil- TiO_2	WCaOT	25.2
Corn Oil- TiO_2	CoOT	24.7
Waste Corn Oil- TiO_2	WCoOT	4.5
Sunflower Oil- TiO_2	SOT	34.7
Waste Sunflower Oil- TiO_2	WSOT	5.7

Table 4 listed all zeta potential measurement for all sample of nanofluid. Specimen of CaOT shows a higher stability and followed by SOT. WCaOT and CoOT shows the same low stability, while WPOT is lower than WCaOT and CoOT. For WCoOT and WSOT is at the lowest level of stability as previously expected. Holistically, as weight of nanofluid is increase, zeta potential measurement increases too. Viscosity of the cooking and waste cooking oil also could influence the stability of the nanofluid and needs more analysis and research on this aspect to increase their stability [22].

3.3 Thermal Conductivity Analysis

By applying KD2 Pro Thermometer on each sample. The respond and the test variable are given in Table 5.

Palm oil shows the same thermal conductivity readings for the 3 samples (PO, POT and WPOT) released. For sunflower oil, 3 samples (SO, SOT and WSOT) showed different reading values but the highest reading was SOT for this group. The reading value of canola oil also showed significant differences for the 3 samples (CaO, CaOT and WCaOT) where CaOT showed the highest reading value for this group. It can be concluded that, for all 12 nanofluid samples CaOT was the highest reading. This proved that the combination of TiO2 with CaOT is required to produce nanofluids with superior thermal conductivity.

Table 5
Value result of thermal conductivity analysis

Oil Type	Sample Labelling	Thermal Conductivity, k (W/mK)
Palm Oil	PO	0.161
Palm Oil- TiO_2	POT	0.162
Waste Palm Oil- TiO_2	WPOT	0.162
Canola Oil	CaO	0.158
Canola Oil- TiO_2	CaOT	0.165
Waste Canola Oil- TiO_2	WCaOT	0.160
Corn Oil	CO	0.158
Corn Oil- TiO_2	CoOT	0.160
Waste Corn Oil- TiO_2	WCoOT	0.160
Sunflower Oil	SO	0.157
Sunflower Oil- TiO_2	SOT	0.160
Waste Sunflower Oil- TiO_2	WSOT	0.160

3.4 Microstructure Analysis

The microstructure that is produced by the high carbon steel consists of ferrite-pearlite structure. Before the evaluation, all specimens will be etched with nital plus 1% zephiran chloride on surface of high carbon steel. The purpose of etching process is to get the preferentially reveals of the microstructure.

Figure 4 shows the microstructure of high carbon steel before heat treatment process, observed under optical microscope. Based on the result shown, fine mixture of ferrite and iron carbide, and pearlite are observed on the surface of high carbon steels specimen.

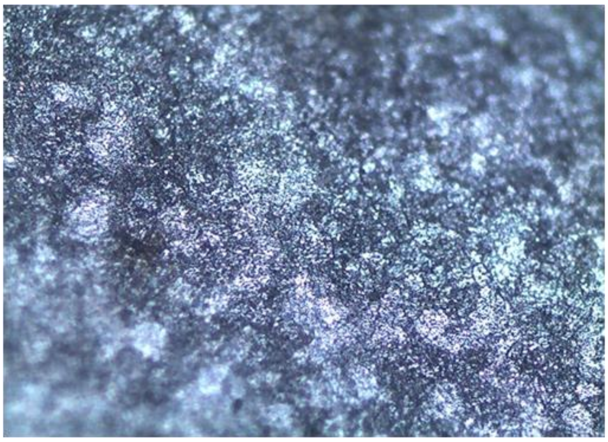


Fig. 4. Microstructure of high carbon steel of magnification with 50X before heat treatment

Figure 5(a) shows the results of high carbon steel by quenching in palm oil (PO) process without TiO_2 while Figure 5(b) and Figure 5(c) from specimen quenched by cooking palm oil (POT) and waste cooking palm oil (WPOT) with TiO_2. Based on the result shown, only a slightly differences on the microstructure of the different quenched sample can be observed as the thermal conductivity of the PO, POT and WPOT are not much changes in value. It can be seen the pearlite can be resolved and the grains are oriented in a direction.

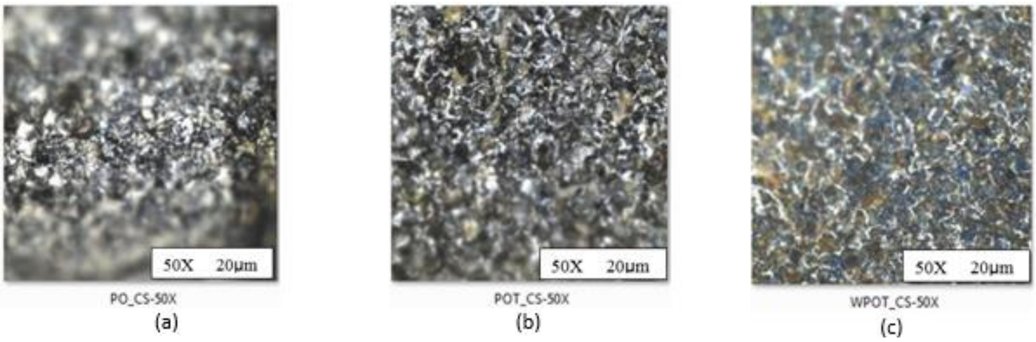


Fig. 5. Microstructure of high carbon steel of magnification with 50X after quenching process on PO, POT and WPOT

Figure 6(a) shows the results of high carbon steel by quenching in canola oil (CaO) process without TiO_2 while Figure 6(b) and Figure 6(c) from specimen quenched by cooking canola oil (CaOT) and waste cooking canola oil (WCaOT) with TiO_2. Based on the result shown, mixture of fine martensite and pearlite are observed on the microstructure of the quenched sample surface.

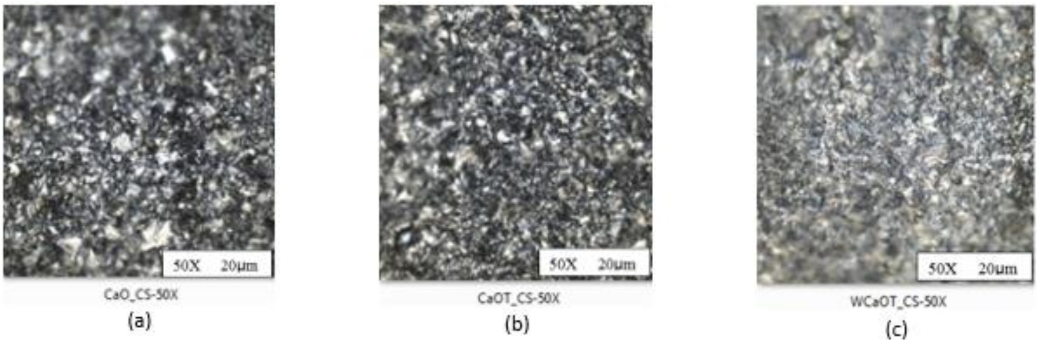


Fig. 6. Microstructure of high carbon steel of magnification with 50X after quenching process on CaO, CaOT and WCaOT

Figure 7(a) shows the results of high carbon steel by quenching in sunflower oil (SO) process without TiO_2 while Figure 7(b) and Figure 7(c) from specimen quenched by cooking sunflower oil (SOT) and waste cooking sunflower oil (WSOT) with TiO_2. Based on the result shown, needle-like structure was observed for SOT quenched sample which attributed to the martensite phases. But cementite and ferrite phases were observed for SO and WSOT quenched sample. This could be due to the low thermal conductivity of SOT and WSOT nanofluid which is used for quenching process.

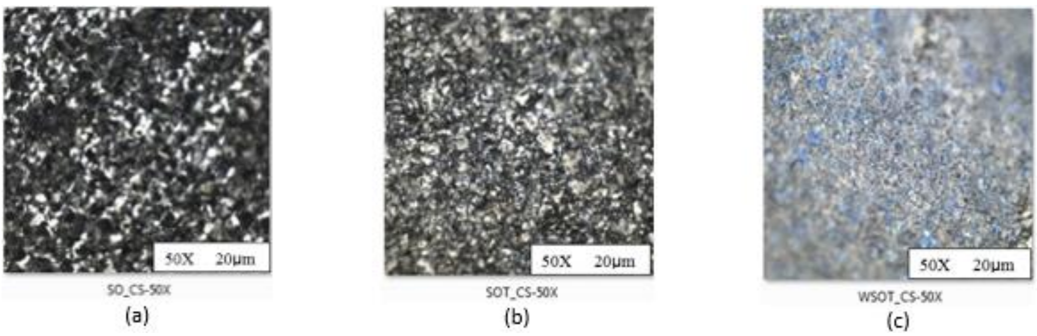


Fig. 7. Microstructure of high carbon steel of magnification with 50X after quenching process on SO, SOT and WSOT

Figure 8(a) shows the results of high carbon steel by quenching in corn oil (COO) process without TiO_2 while Figure 8(b) and Figure 8(c) from specimen quenched by cooking corn oil (CoOT) and waste cooking corn oil (WCoOT) with TiO_2. Based on the result shown, fine unresolved pearlites are observed on the microstructure of quenched sample surface.

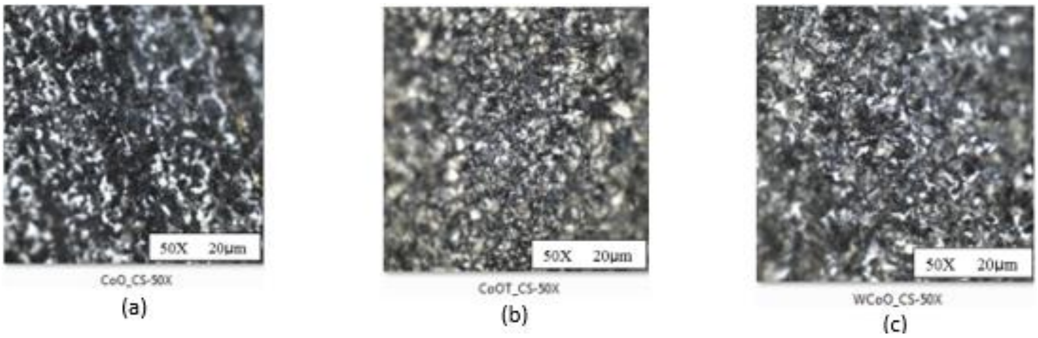


Fig. 8. Microstructure of high carbon steel of magnification with 50X after quenching process on CoO, CoOT and WCoOT

3.5 Hardness Test

Table 6 and Figure 9 represent the average hardness value of the high carbon steel samples. The Vickers hardness values samples quenched in POT, SOT and CaOT are higher than other all specimens that are tested. Only COT showing a reverse trend of hardness value. It is in agreement of the thermal conductivity value of the COT compared to that of other cooking oil based nanofluid (POT, SOT and CaOT). Due to lower thermal conductivity of the COT, the martensite phase transformation is not happened during the quenching process leading to lower hardness value. Hardness value of the quenched sample in POT and CaOT show the highest value which are in agreement with the result obtained in thermal conductivity of those samples. The hardness value is linear to its microstructure which shows the most martensite structure.

This result shows that the TiO_2 nanoparticles inside the oil base fluid can enhance the fluid severity. This is happened due to the transformation of martensite with rapid cooling. Transformation of martensite is driven by TiO_2 nanoparticles in the oil-based fluid which is having higher thermal conductivity than in normal oil fluid. The enhancement of heat transfer can be explained by the formation of nanoparticle embedded surface due to scattered deposition of TiO_2 nanoparticles. This would lead to the increases of the effective surface area of heat transfer.

Table 6
Type of oil base fluid

Sample oil	CS
PO	253.67
POT	306.67
WPOT	252.67
SO	190.67
SOT	226.00
WSOT	176.00
CaO	229.67
CaOT	307.67
WCaOT	205.67
CoO	191.33
CoOT	180.67
WCoOT	199.40

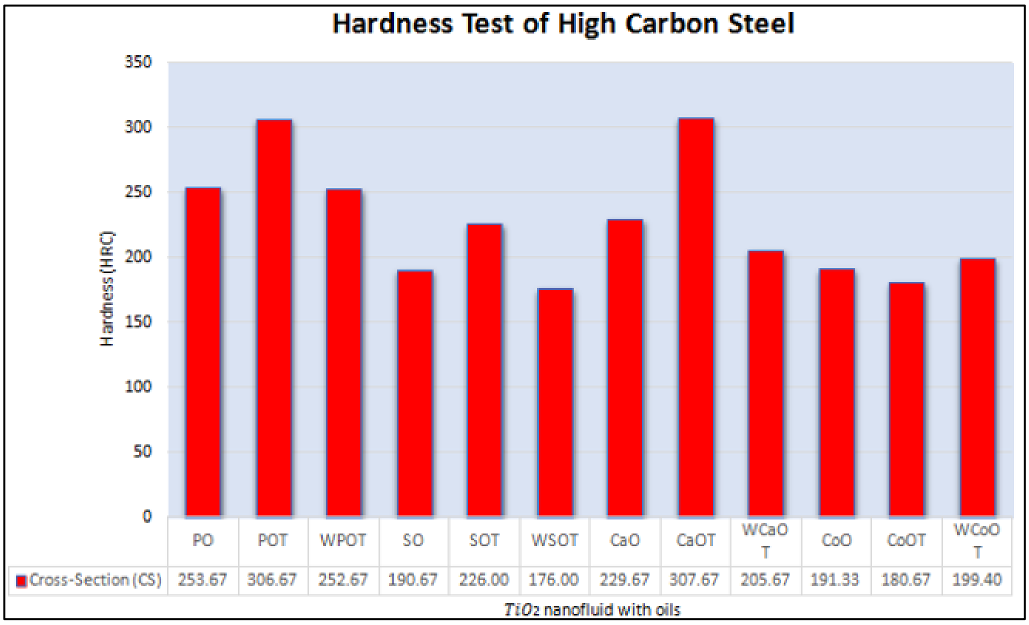


Fig. 9. Graph of hardness test on high carbon steel

4. Conclusions

In this study, after synthesising nanofluid with different cooking and waste cooking oil as a base fluid, the results are proved qualitatively and quantitatively by sedimentation method and zeta potential measurement respectively. Adding nanoparticles in oil base fluid will enhance stability, thermal conductivity and improve thermal performance of heat transfer of the base fluid.

Nanofluids have been receiving great attention in recent years due to their potential usage, not only as an enhanced thermophysical heat transfer fluid but also because of their great importance in applications such as affected the quenching process. The effect of laboratory grade carbon in oil as quench medium was experimentally investigated using high carbon steel as specimen. For this purpose, several of nanofluids with cooking and waste cooking oil were prepared with two step method. The microstructure and hardness value shows that difference oil of nanoparticles used in nanofluids considerably affected the quenching process. Quenching with oil-based nanofluid will produce martensite structure.

The Vickers hardness values of POT and CaOT are higher than other all specimens that are tested. This is because both of specimens it consists of ferrite and martensite structures and better hardness properties. Observation nanofluid with cooking and waste cooking oil also can act as chemical stability with the mass production thus it is easy to get because it is economical.

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