Optimization of Sodium Dodecylbenzene Sulfonate Surfactant and Dispersed Coconut-Shell Carbon Particles to the Thermal Conductivity of Heat Treatment Quenchant for S45C Medium Carbon Steel

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Abstract: Dispersing stabilized carbon-based solid particles in a base fluid can increase its thermal conductivity. The higher thermal conductivity of the carbon particles compared to the base fluid increases its heat transfer characteristics. A practical example of this type of thermally enhanced fluid for use as a quench medium in the steel heat treatment industry is presented here. By controlling the amount of carbon-based dispersed particles and stabilizers added to quench mediums, the cooling rate of the quench medium can be altered, which affects steel hardness after heat treatment. This study synthesized quenchants using distilled water; 0.1, 0.3, and 0.5 wt% dispersed coconut-shell charcoal carbon particles; and 3%–30% sodium dodecylbenzene sulfonate (SDBS) as a surfactant. The quenchants were used to quench S45C medium carbon steel samples. The highest thermal conductivity (0.74 W/mK) and highest steel hardness (55 HRC) were achieved with the addition of 3% SDBS. Increased steel hardness correlated with higher cooling rates due to the increased thermal conductivity of the quenchant.

Keywords: Sodium Dodecylbenzene Sulfonate, Coconut-Shell Carbon, Thermal Conductivity, Quenchant, S45C Steel.

1. Introduction

Quenching is a crucial process for the heat treatment of steel, as it can enhance the steel's mechanical properties, especially its hardness. In this process, steel is heated to elevated temperatures to reach the austenite structure phase. The steel is then rapidly cooled or quenched to room temperature. During rapid cooling, the austenite phase transforms into the martensite phase, which has a very high hardness [1]. The most crucial parameter of the quenching process is the cooling rate because too slow a cooling rate hinders martensite phase formation.

However, if the cooling rate is too fast, there is a risk of steel cracking or distorting. Hence, an appropriate quench-medium cooling rate is critical [2]. Furthermore, suitable cooling rates vary according to the type of steel. In addition, adding solid particles to a quench medium can alter its thermal conductivity, which alters its cooling rate [3]. Therefore, the cooling rate of the quench medium can be controlled as required. The effects of the addition of solid particles to quench mediums have been widely studied [4, 5, 6, 7]. Several types of solid particles, such as metal particles, metal oxides, and carbon-based particles, are added to quenchants to increase their thermal conductivity [8, 9, 10]. The use of carbon-based particles, such as graphene, graphene oxide, and carbon nanotubes, is of interest because of their exceptionally high thermal conductivity [11, 12]. However, they are costly. Alternatively, bio-based carbon particles, commonly called carbon biomass, are inexpensive and abundant. The use of coconut charcoal particles as biomass dispersants in quench mediums is well known [13, 14, 15].

The main challenge to particle-added fluids is agglomeration because agglomeration in a quench medium can reduce the effectiveness of solid particle heat transfer. Because coconut charcoal particles are likely to agglomerate in quench mediums (i.e., water), surfactants are commonly added to improve their dispersion [16, 17]. Surfactants modify the surface tension of particles, which makes them easier to disperse [18], and better particle dispersion increases the thermal conductivity of quench mediums.

2. Experiment Setup

Commercially available coconut-shell charcoal biomass was used for the carbon particles. To reduce the particle size, the charcoal was milled in a planetary ball mill (NQM-4 L, from Shenzen, China) for 15 hours with a jar rotation speed of 500 rpm. A stainless-steel jar and grinding ball were used to contain and reduce the size of the carbon particles. This study used a typical 10:1 ball-to-powder ratio for the milling process. Field-emission scanning electron microscopy (SEM; Inspect F-50) with integrated energy dispersive x-ray analysis (EDAX) was used to characterize the morphology and chemical composition of the particles, respectively, in this research. A particle size analyzer (PSA; Horiba SZ-100) using the dynamic light scattering method was used to determine the particle size and shape before and after milling. After milling, the carbon particles were used to synthesize carbon particle-dispersed fluids using distilled water as the base fluid and 0.1, 0.3, and 0.5 wt% carbon particles, respectively. A surfactant, sodium dodecylbenzene sulfonate (SDBS), was then added to the carbon particle-dispersed fluids to increase their stability. This study investigated the use of 3%–30% SDBS in each of the dispersed-particle fluid variations. The thermal conductivity of each fluid was characterized using a thermal properties analyzer (KD2). The Horiba SZ-100 was also used to determine the fluid stability of each fluid using zeta potential. The fluids were then used as experimental heat treatment quenchants after sonification for 15 min in a bath-type ultrasonicator.

S45C medium carbon steel was used for the heat treatment process. The composition of the S45C medium carbon steel was checked using optical emission spectroscopy (OES; Foundry-Master Xpert). Before annealing the steel samples, preheating was conducted at 540°C for 10 min. Preheating is vital to avoid cracking due to temperature differences between the core and surface of the steel. The annealing was undertaken at 900°C for 60 min to achieve the complete austenite phase. Subsequently, the steel was quenched in the synthesized fluids.

An optical microscope and a Rockwell hardness machine were used to characterize the quenched steel samples by checking their microstructure and hardness. A 2% nital solution was used to expose the microstructure of the steel, which was then observed using an inverted metallurgical microscope (BX41M-LED; Olympus). The hardness of the original and heat-treated steel samples was determined using a digital hardness tester (Qualirock), implemented using the ASTM E18 standard for Rockwell hardness (HRC) testing.

3. Result and Discussion

3.1 Coconut-Shell Carbon Particle

The as-received coconut-shell carbon particles were clumped together and irregularly sized, as shown in Figure 1a. Particle size measurements indicated that the average particle size was 1.147 × 104 nm. After ball milling, the particles were visually smaller; however, the particles were agglomerated, as shown in Figure 1b. The average particle size after milling was 1,762 nm. The chemical composition of the biomass particles, determined using EDS, indicated that they were composed of carbon and several impurities, such as sodium, magnesium, and other elements, as shown in Table 1.

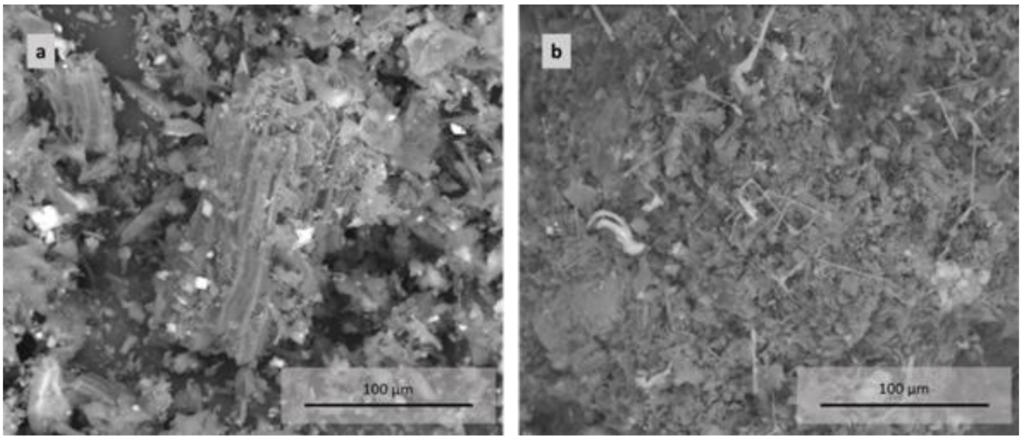

Figure 1. *Scanning electron microscope (SEM) micrograph of coconut-shell carbon particles. (a) before milling and (b) after milling.*

Table 1. *Coconut-shell carbon particle composition by EDS*

Element	Weight %	Atomic %
C	64.27	73.0
O	25.51	27.75
Na	3.74	2.22
Mg	1.20	0.67
Al	1.52	0.77
Si	1.64	0.80
Cl	1.28	0.49
K	0.84	0.29

Source: Authors

3.2 Carbon Particle-Dispersed Fluid Characterization

SDBS surfactant was added to the carbon particle-dispersed fluids to prevent agglomeration and increase stability [19, 20]. A particle dispersion stability test using zeta potential was conducted on the fluid sample with 0.5 wt% particles, and the results are shown in Table 2. It was assumed that a higher SDBS content would improve particle stability, meaning that the particles would be dispersed for longer in the fluid without precipitating.

However, adding 20% and 30% SDBS decreased the zeta potential. This compromises particle dispersion because the excessive addition of surfactant can result in the formation of micelles, which reduces the effectivity of surfactants to modify the particle surface tension. It has been reported that micelles can have a solubilization effect on surfactant molecules, which decreases the adsorption strength of particles [18].

Another study showed that excess surfactant reduces the net charges of particles, which results in weak dispersion [20]. In addition, another study showed that an increase in surfactants may result in flocculation, which reduces the suspension of particles [21]. Overall, increased amounts of surfactant lower fluid stability.

Table 2. *Zeta potential analysis of carbon particle-dispersed fluid stability*

Carbon content (%)	SDBS content (%)	Zeta potential (mV)
0.5	0	- 20.1
	3	- 50.3
	5	-51.5
	7	-53.4
	10	-53
	20	-46.2
	30	-37.8

Source: Authors

Figure 2 shows the results of the thermal conductivity testing. The optimum surfactant addition was 3%. In this fluid variant, the thermal conductivity reached 0.74 W/mK. This is higher than the thermal conductivity of distilled water without the addition of any particles or surfactant (0.61 W/mK). The lowest thermal conductivity (0.44 W/mK) was obtained for the fluid sample containing 30% surfactant. As explained, higher percentages of added surfactant can form micelles, which reduces particle stability. Lower stability means that particles tend to agglomerate more rapidly, which can result in an inhomogeneous quench medium. Therefore, the thermal conductivity is also reduced. However, different percentages of carbon particles in the quenchant may have insignificant effects on the thermal conductivity of each surfactant variation. This insignificant result may be due to the carbon particle's size not being small enough to increase the thermal conductivity with increasing particle content.

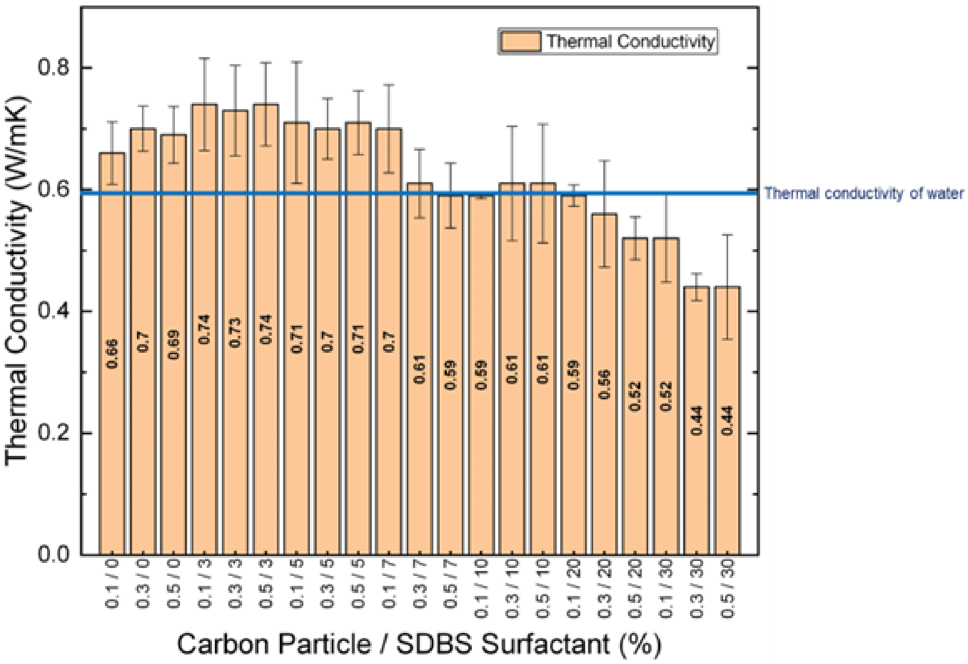

Figure 2. *Thermal conductivity of carbon particle-dispersed fluids with sodium dodecylbenzene sulfonate (SDBS) surfactant.*

3.3 Characterization of S45C Steel

The results of the OES characterization of the chemical composition of the S45C steel are shown in Table 3. Figure 3 shows the microstructure of a sample of the steel before heat treatment. As expected, the microstructure consisted of only ferrite and pearlite phases. The result of the steel hardness test also supported the microstructure type, as the steel hardness was only 12 HRC. Steel samples were then heat-treated by annealing and quenching using the variously prepared carbon particle-dispersed fluids. The heat treatment profile is presented in Figure 4.

Table 3. *Medium carbon steel S45C composition*

Element	%
Fe	98.2
C	0.425
Si	0.264
Mn	0.68
P	0.008
S	0.006

Source: Authors

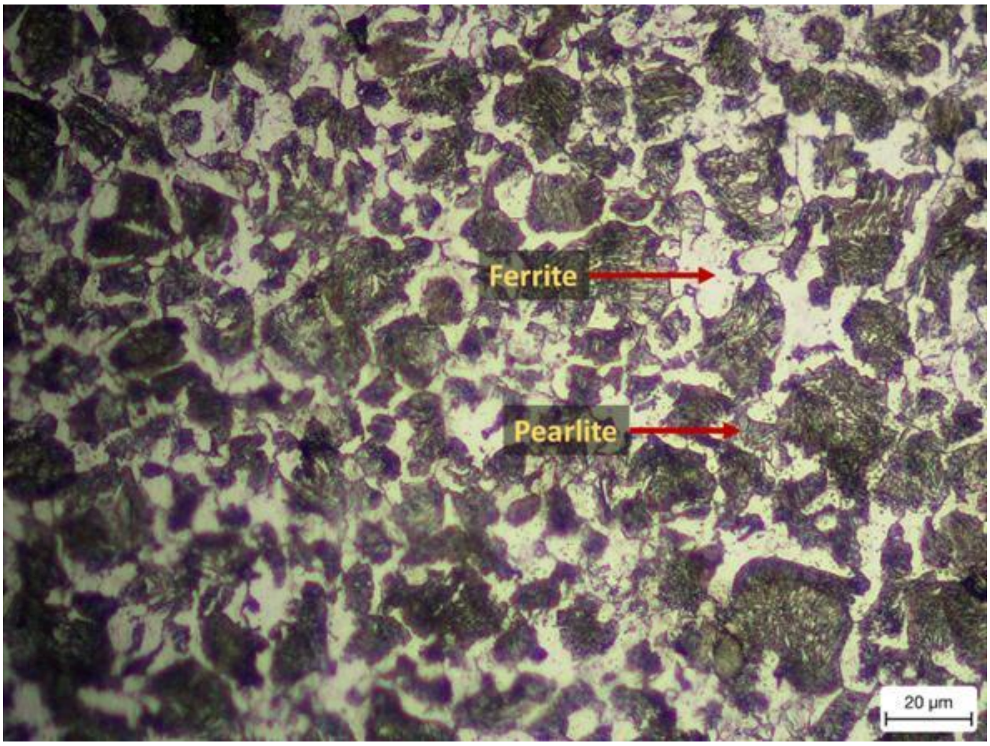

Figure 3. *Nital-etched microstructure of the S45C medium carbon steel before heat treatment.*

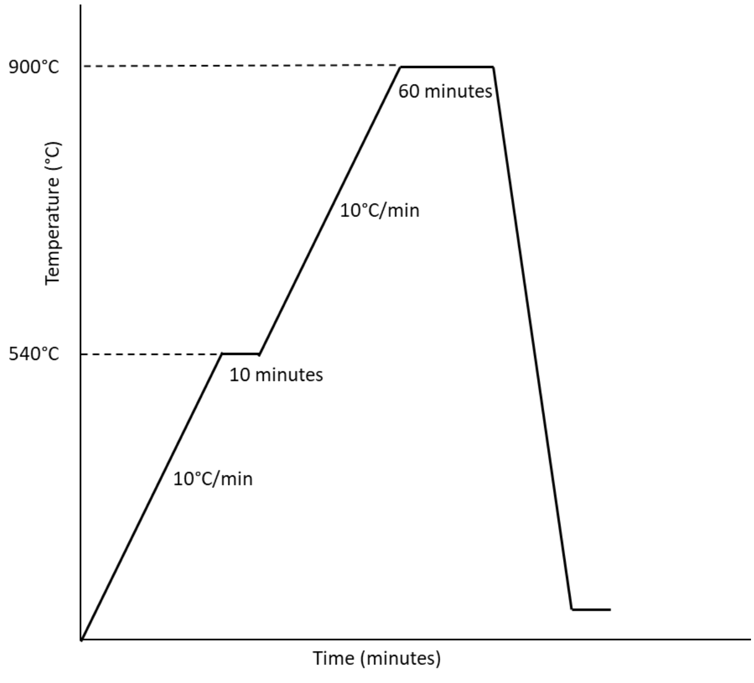

Figure 4. *Heat treatment profile for the S45C medium carbon steel sample.*

The first steel sample was quenched in only distilled water to compare the effects of adding carbon particles and surfactant. The microstructure of the water-quenched sample is shown in Figure 5. Because of rapid cooling, the microstructure consisted of bainite and martensite phases. The hardness value of the steel increased to 43 HRC.

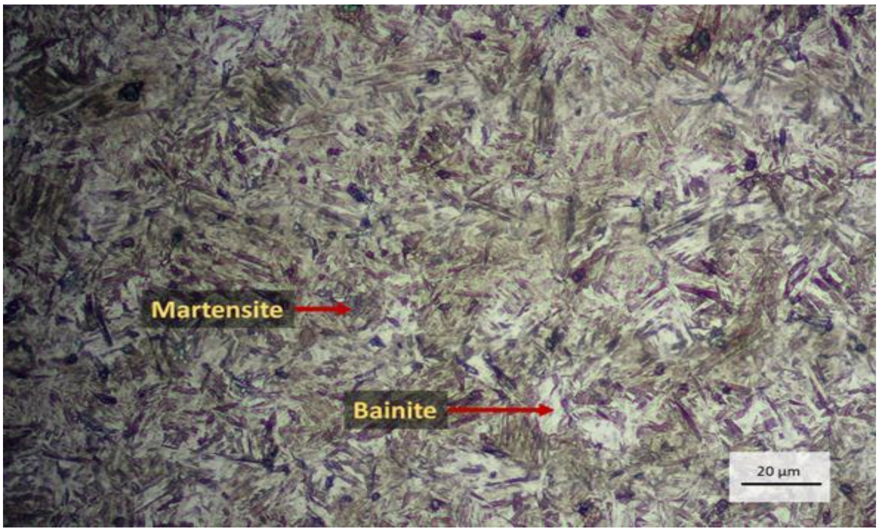

Figure 5. *Nital-etched S45C medium carbon steel microstructure after quenching with distilled water.*

The other samples were then quenched using quenchant fluids containing the variable amounts of carbon particles and surfactant. Figure 6 shows the hardness of the steel samples after quenching with the different quenchants. The highest hardness was 55 HRC for the sample quenched in 0.1% carbon and 3% SDBS. This was a roughly 28% improvement in HRC hardness number compared to the steel samples quenched in distilled water.

The results of the post-quench hardness testing were in line with the results of the thermal conductivity testing. Higher thermal conductivity results in faster cooling rates and, hence, higher steel hardness. Therefore, for example, a sample quenched in a quenchant containing more surfactant results in lower steel hardness due to the lower thermal conductivity of the quenchant. Therefore, it can be concluded that the thermal conductivity of a quenchant affects the cooling rate and quenched steel hardness.

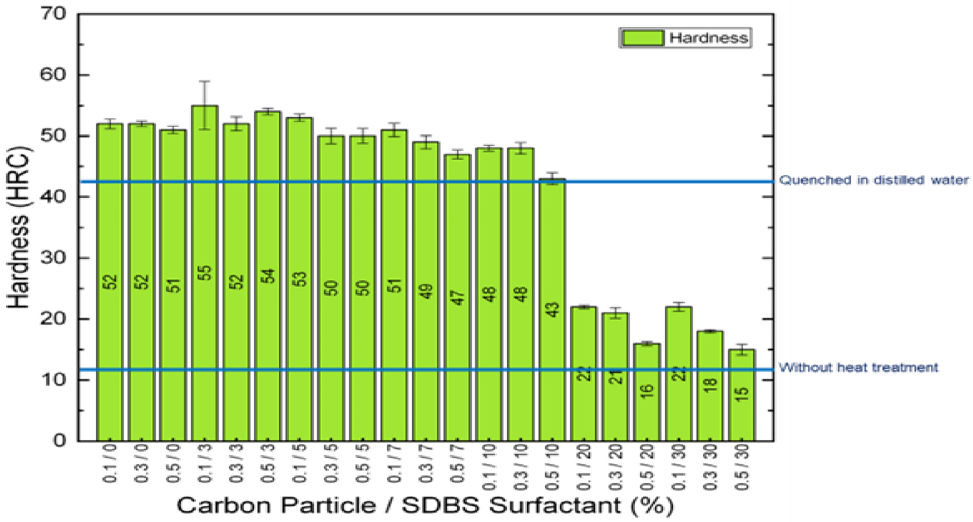

Figure 6. *Hardness of S45C medium carbon steel samples quenched in different carbon-particle dispersed quenchant with sodium dodecylbenzene sulfonate (SDBS) surfactant.*

Microstructural observations were conducted to support the measured hardness of the steel samples. The samples were ground, polished, and etched using a 2% nital solution. Figures 7–13 show the microstructures of the steel samples after quenching with the various quench fluids. Most of the samples after quenching contained martensite, bainite, and a small number of ferrite phases. However, steel samples quenched in quench mediums containing 20% or more SDBS contained only combinations of ferrite and pearlite phases. Therefore, the microstructural results supported the hardness results. Bainite and martensite provide higher hardness, while ferrite and pearlite provide much lower hardness.

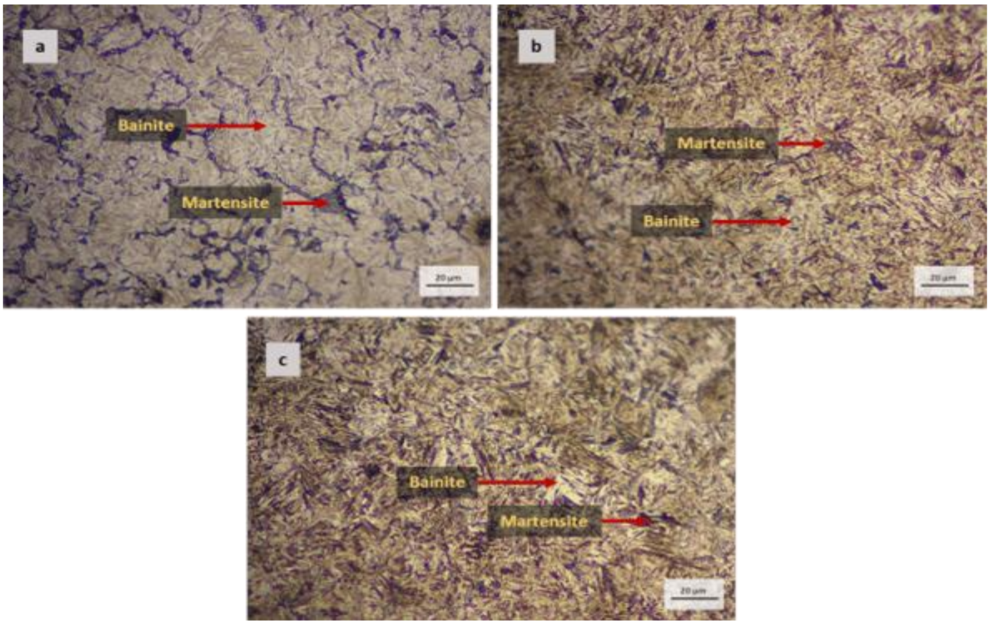

Figure 7. *Nital-etched microstructure of S45C medium carbon steel samples quenched without sodium dodecylbenzene sulfonate surfactant. Carbon particles: (a) 0.1wt%; (b) 0.3wt%; and (c) 0.5wt%.*

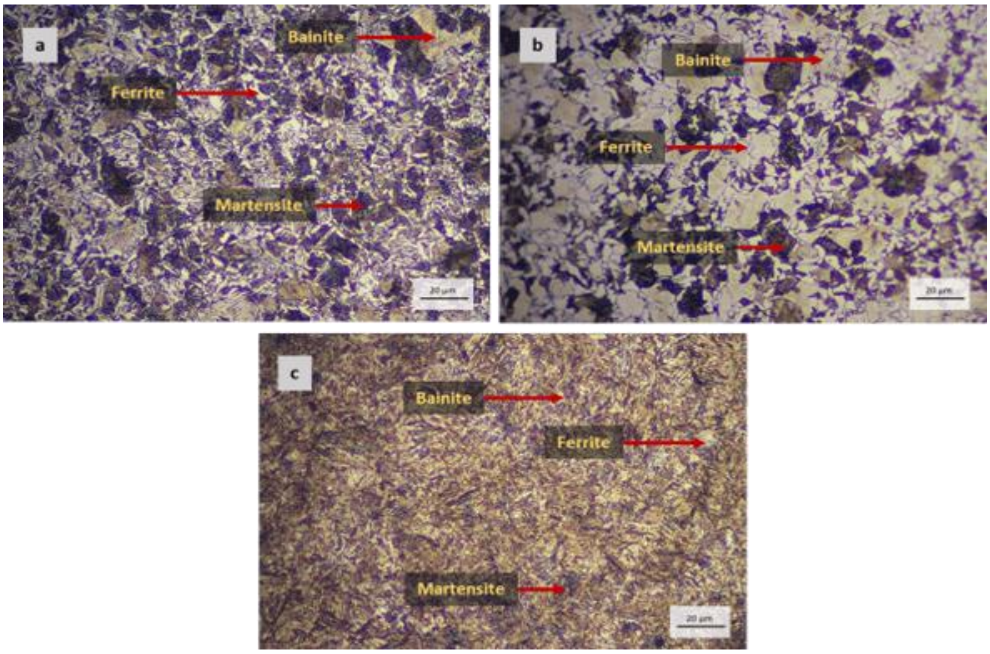

Figure 8. *Nital-etched microstructure of S45C medium carbon steel samples quenched with 3% sodium dodecylbenzene sulfonate surfactant. Carbon particles: (a) 0.1wt%; (b) 0.3wt%; and (c) 0.5wt%.*

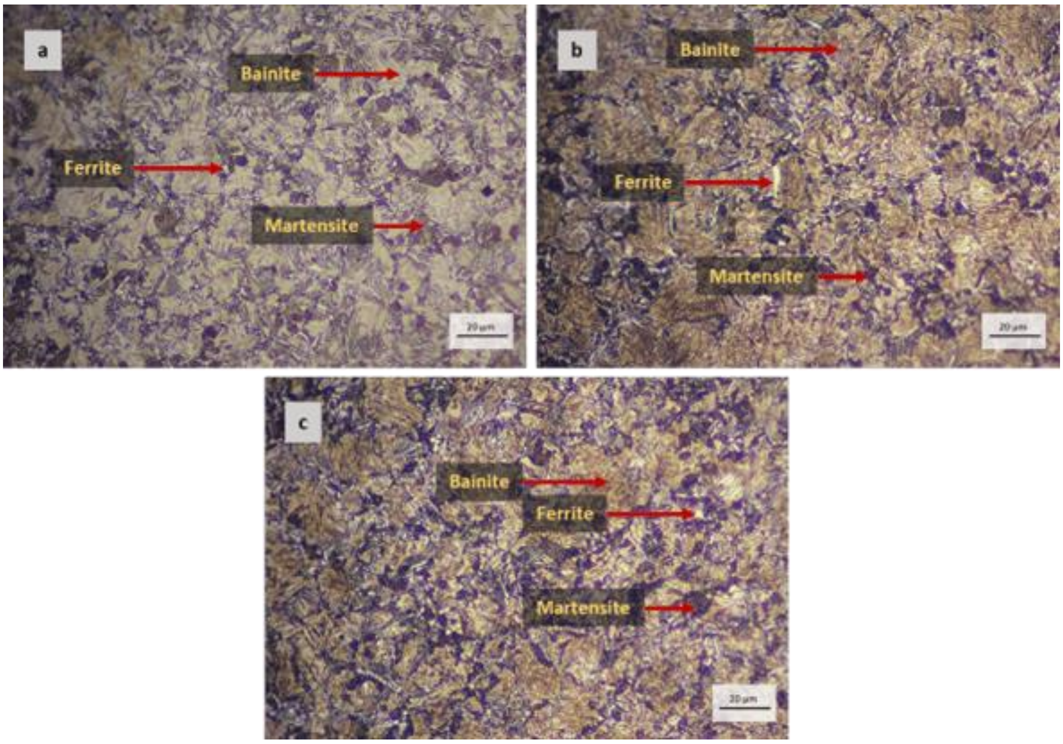

Figure 9. *Nital-etched microstructure of S45C medium carbon steel samples quenched with 5% sodium dodecylbenzene sulfonate surfactant. Carbon particles: (a) 0.1wt%; (b) 0.3wt%; and (c) 0.5wt%.*

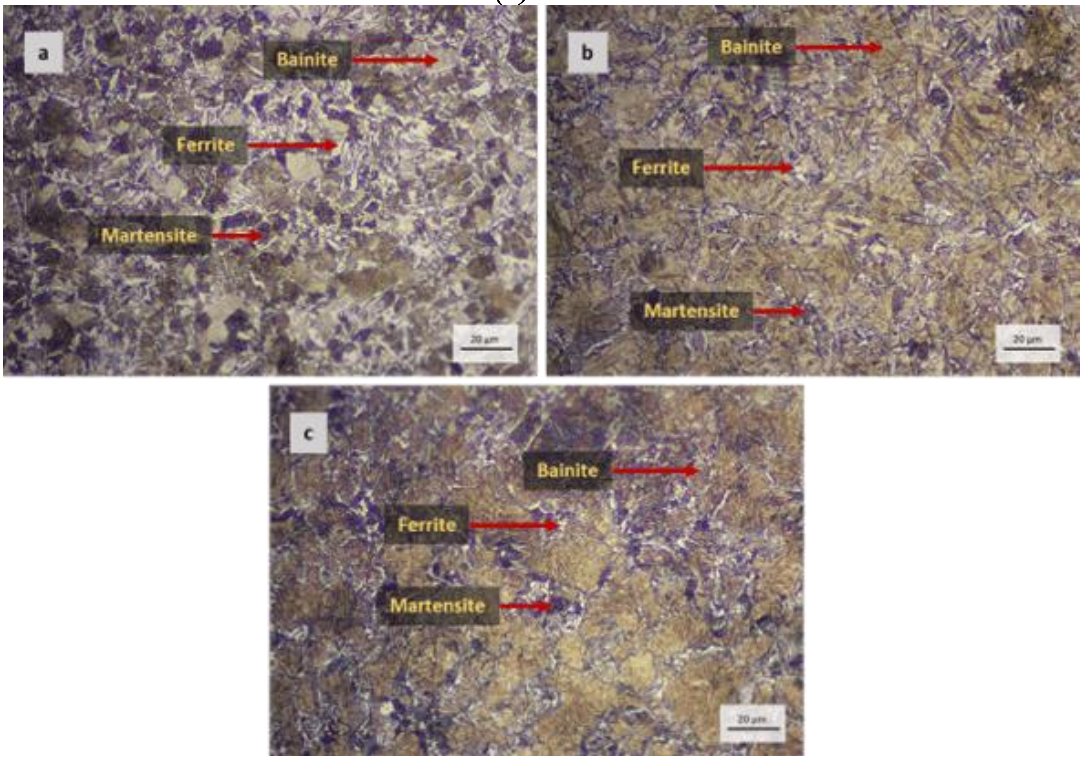

Figure 10. *Nital-etched microstructure of S45C medium carbon steel samples quenched with 7% sodium dodecylbenzene sulfonate surfactant. Carbon particles: (a) 0.1wt%; (b) 0.3wt%; and (c) 0.5wt%.*

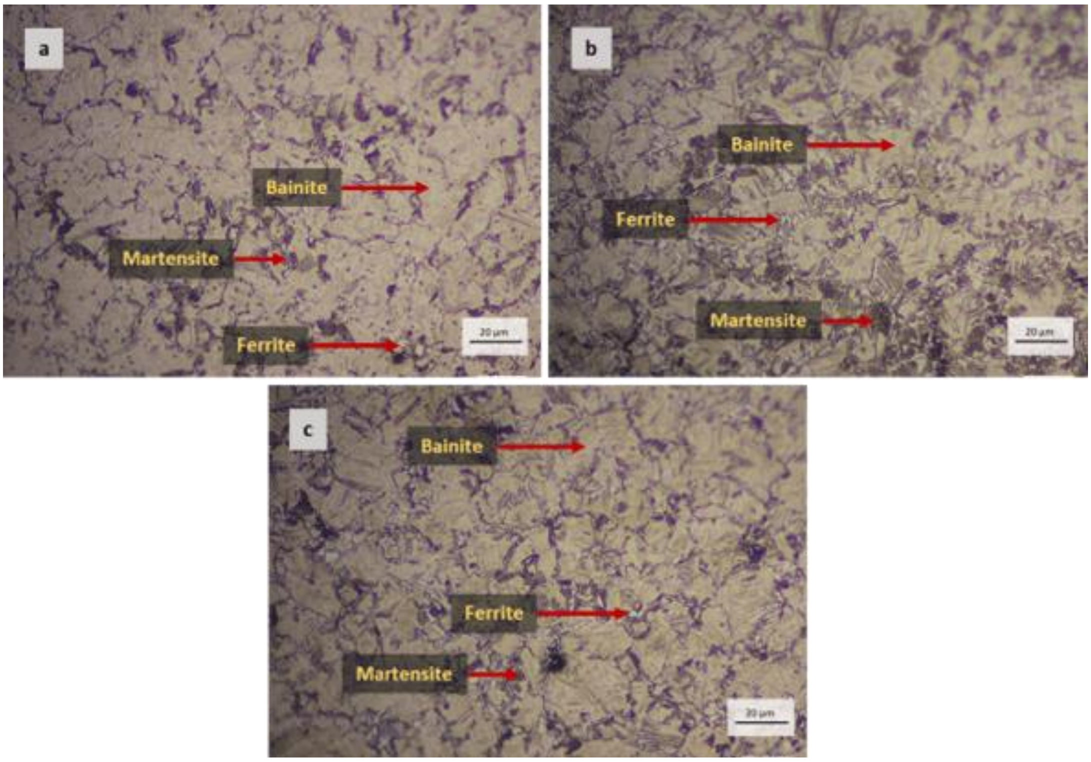

Figure 11. *Nital-etched microstructure of S45C medium carbon steel samples quenched with 10% sodium dodecylbenzene sulfonate surfactant. Carbon particles: (a) 0.1wt%; (b) 0.3wt%; and (c) 0.5wt%.*

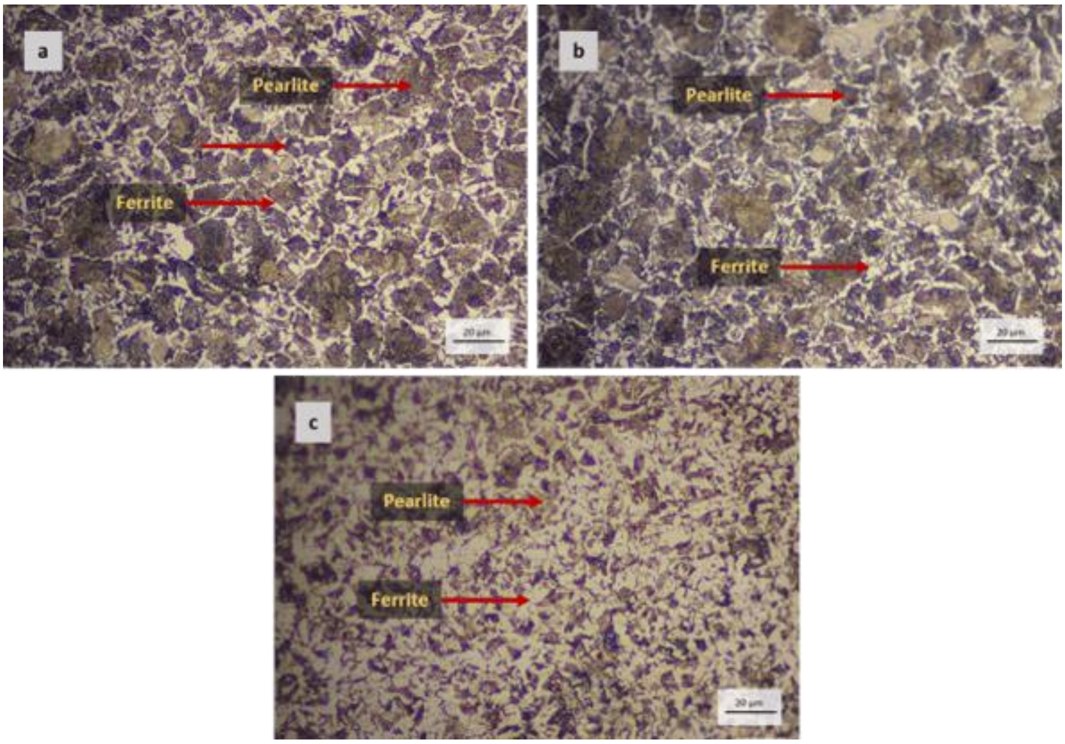

Figure 12. *Nital-etched microstructure of S45C medium carbon steel samples quenched with 20% sodium dodecylbenzene sulfonate surfactant. Carbon particles: (a) 0.1wt%; (b) 0.3wt%; and (c) 0.5wt%.*

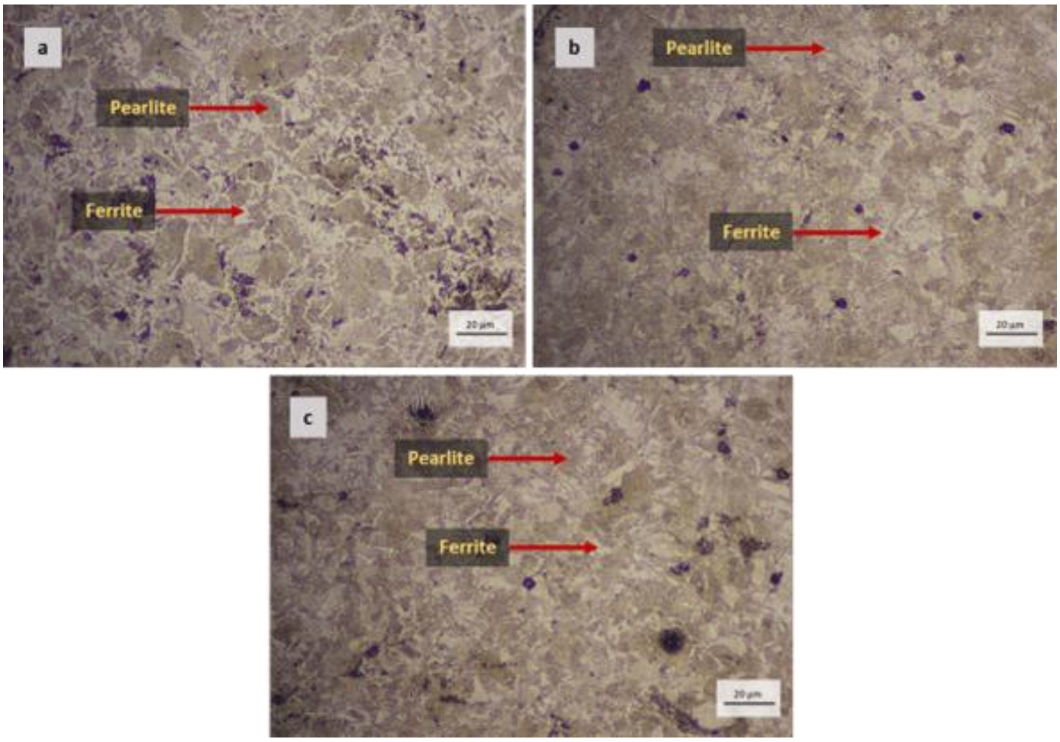

Figure 12. *Nital-etched microstructure of S45C medium carbon steel samples quenched with 30% sodium dodecylbenzene sulfonate surfactant. Carbon particles: (a) 0.1wt%, (b) 0.3wt%, and (c) 0.5wt%.*

Furthermore, the results of the microstructural observations were aligned with the thermal conductivity results: higher surfactant percentages resulted in decreased thermal conductivity. Lower thermal conductivity can slow the cooling rate; hence, the ferrite and pearlite phases have lower hardness.

Phase calculations were conducted to further investigate and compare the microstructural phases. ImageJ software was used to calculate the phase percentages of each sample. Table 4 shows the results of the calculations.

Table 4. *Phase percentage calculation*

Variables (%carbon / %surfactant)	**Ferrite (%)**	**Pearlite (%)**	**Bainite (%)**	**Martensite (%)**	**Hardness (HRC)**
0,1 / 0	0	0	89	11	52
0,3 / 0	0	0	87	13	52
0,5 / 0	0	0	87	13	51
0,1 / 3	9	0	30	61	55
0,3 / 3	12	0	43	45	52
0,5 / 3	8	0	27	65	54
0,1 / 5	10	0	57	33	53
0,3 / 5	9	0	61	30	50
0,5 / 5	10	0	59	31	50
0,1 / 7	23	0	42	35	51
0,3 / 7	17	0	56	27	49
0,5 / 7	16	0	59	25	47
0,1 / 10	15	0	73	12	48
0,3 / 10	16	0	69	15	48
0,5 / 10	22	0	70	8	43
0,1 / 20	43	57	0	0	22
0,3 / 20	43	57	0	0	21
0,5 / 20	45	55	0	0	16
0,1 / 30	42	58	0	0	22
0,3 / 30	44	56	0	0	18
0,5 / 30	47	53	0	0	15

Source: Authors

The steel sample quenched in the carbon particle-dispersed quench medium containing no SDBS resulted in more bainite than martensite, indicating slightly lower thermal conductivity and a slower cooling rate. Adding 3% SDBS improved the thermal conductivity and increased the martensite phase, indicating a higher cooling rate and, hence, higher steel hardness. However, adding 5%, 7%, and 10% SDBS lowered the thermal conductivity and decreased the cooling rate. This reduced the number of martensite and bainite phases and increased the ferrite phase. Higher percentages of SDBS added to the quench medium further expanded the ferrite and pearlite phases, especially after the addition of 20% and 30% SDBS. Correspondingly, the steel hardness was lower. Again, the decrease in hardness may have been due to a slower cooling rate due to the lower thermal conductivity of the quenchants containing higher percentages of SDBS.

It was concluded that the cooling rates of the quench mediums were related to thermal conductivities. Figure 13 shows the relationship between the thermal conductivity of the quench medium and steel hardness.

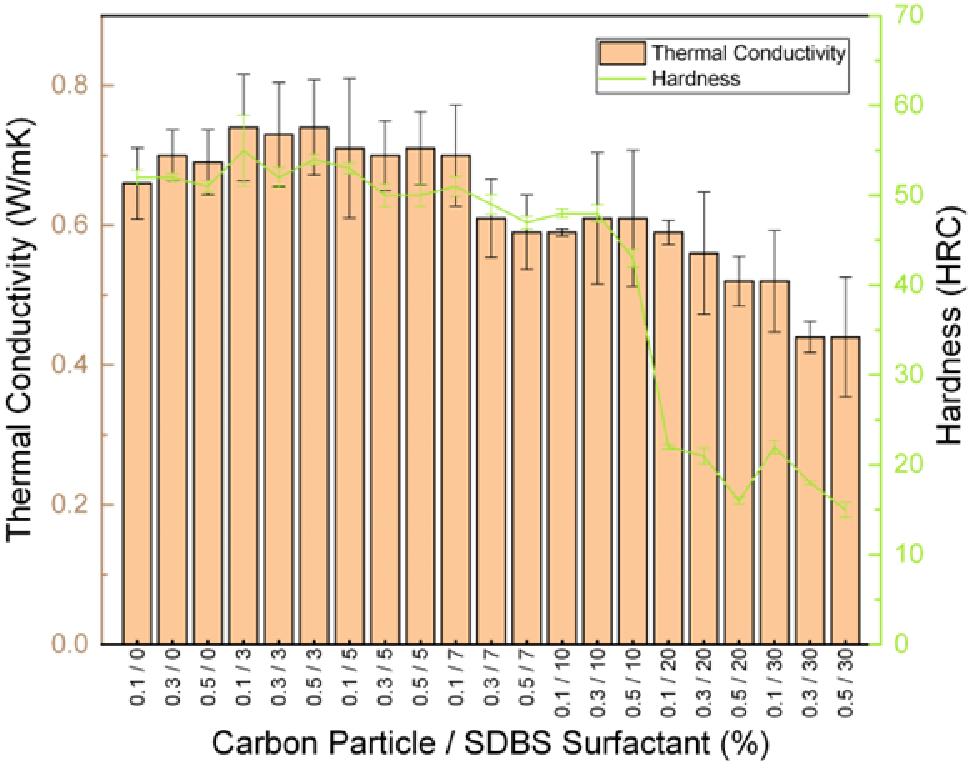

Figure 13. *Relationship between the thermal conductivity of carbon particle–dispersed fluid quenched medium and steels hardness.*

Quench mediums containing excessive surfactant appeared to significantly decrease the hardness of the steel samples. Two factors may have caused this apparently nonlinear relationship. The first is micelle formation, as explained in Section 3.2. The second is that the surfactant had lower thermal conductivity compared to distilled water. The measured thermal conductivity of a saturated solution of SDBS (0.22 W/mK) was much lower than that of water (0.61 W/mK).

Another possible reason is that the higher percentages of surfactant increased the viscosity of the quench medium. In particle-dispersed fluids, higher viscosities can reduce Brownian motion, which is required to increase thermal conductivity. Higher viscosities decrease thermal conductivity and cooling rates more significantly [22].

4. Conclusion

A carbon particle-dispersed quench medium made using coconut-shell charcoal particles was synthesized. The particles were milled in a planetary ball mill to reduce the average particle size from 1.147 × 104 nm to 1,762 nm. The particles (0.1, 0.3, and 0.5 wt%) were then added to distilled water to synthesize various quench mediums. The addition of the particles increased the thermal conductivity of the quench medium to 0.7 W/mK. SDBS was then added as a surfactant to increase the stability of the quench mediums. The surfactant helped to further improve the thermal conductivity of the quench medium to 0.74 W/mK. The highest thermal conductivity was obtained with the addition of 3% SDBS.

Excessive surfactant appeared to decrease the thermal conductivity to 0.44 W/mK. This may have occurred because the formation of micelles reduces the surface tension of the particles. SDBS has a thermal conductivity lower than distilled water; therefore, the addition of high surfactant percentages resulted in very low quench-medium thermal conductivities. Increasing the viscosity of the quench medium may also have further decreased the thermal conductivity.

The steel hardness trends after quenching in the carbon particle-dispersed quench medium were similar. The highest steel hardness (55 HRC) was obtained with the addition of 3% SDBS. The hardness was slightly lower with the addition of 5% SDBS, and it further decreased with higher SDBS percentages. At 30% SDBS, the maximum hardness of the steel was only 22 HRC. This low hardness showed that the lower thermal conductivity of the quench medium affected the cooling rate. More importantly, as shown in this research, the ratio between the dispersed particles and the surfactant plays a critical role in achieving the intended steel hardness.

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