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Research Paper

HEAT TRANSFER CHARACTERISTICS OF WATER BASED EGGSHELL POWDER DURING QUENCHING OF MEDIUM CARBON STEEL

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The potential of water-based eggshell powder (agitated) as a quenching medium for hardening medium carbon steel (SAE-AISI 1045) was investigated. Addition of 25 wt% eggshell powder to water was used to form the water-based eggshell powder quenchant. The water-based eggshell powder quenchant was used in unagitated and agitated conditions in hardening medium carbon steel. Scanning electron microscope and x-ray diffractometer were used to characterize the crystallinity of eggshell powder. The chemistry, specific latent heat of vaporization and cooling rates of the water-based eggshell powder quenchant were determined. The cooling rate of the medium carbon steel quenched in the water-based eggshell powder quenchant was compared to the cooling rates of samples quenched in water and engine oil (SAE40) quenchants. The cooling rate curves were used to determine quench severity. The results obtained show that water-based eggshell powder quenchant has higher specific latent heat of vaporization than water. The water-based eggshell powder quenchant (agitated) gave the highest cooling rate of 225°C/s at 500°C.

Keywords: Heat transfer, Quenching media, Eggshell powder, Latent heat, Cooling rate

INTRODUCTION

Quenching is one of the most important processes of heat treatment which can be used to enhance the performance of steel greatly. Quenching is performed by rapidly cooling from the austenitizing temperature to room temperature to obtain martensite structure, thereby preventing the formation of ferrite and pearlite (Rajan *et al.*, 1988). The main requirement of the quenching medium is to cool the component fast enough to produce the required structure. During quenching, heat is extracted from the material at a very fast rate, which shoulld be equal to or faster than the critical cooling rate. For specific steel composition and heat treatment, there is a cooling rate known as the critical cooling rate for the full hardening of steel. The mechanism of quenching and the factors affecting the process influence quenchant selection and performance. The rate of cooling in the part depends on the heat removal characteristics of the cooling medium, the thermal

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characteristics of the alloy, and the section thickness of the part (Rajan *et al*., 1988).

A useful way of accurately describing the complex mechanism of quenching is to develop a cooling curve for the liquid quenchant under control condition. The temperature-time curve reveals the heat transfer characteristics of the quenching medium. The shape of the cooling curve is representative of the various stages of the cooling mechanism during the quenching process. Three stages are involved, namely the vapour blanket stage, nucleate boiling stage and the convection cooling stage. The ideal quenching medium is one that would exhibit high initial quenching speed in the critical hardening range of vapour blanket and nucleate boiling, and a slow final quenching speed through the lower temperature range of convection cooling. This allows stress equalization, and the reduction of distortion and cracking. The first criterion that any quenchant must meet is its ability to approach this ideal quenching mechanism (Herring and Balme, 2007).

The mechanical strength of medium carbon steels can be improved by quenching in appropriate medium. Some of the widely used quenching media for steels are water, oil, molten salts and polymer solutions. Brine and water, has by far the fastest cooling rate, followed by polymer solutions and finally oil. Water produces more severity of quench than oil. But has a high tendency of developing cracks or distortion in the component. Water soluble oil or polymer can be added to water to lower the cooling rate. The cooling rate of water can also be reduced by raising the initial temperature of the water. If about 10% sodium hydroxide is mixed with water it will have a cooling rate that will be about twice that of water at room temperature (Budinski, 1999). Water is one of the most commonly used quenching medium because it is abundant, inexpensive, easy to handle and has little disposal considerations. It is also one of the fastest quenching media, second only to aqueous solution. (Rajan *et al.*, 1988). It can produce high hardness values and excellent mechanical properties.

Water can be used successfully for certain carbon steels, some alloy steels and non ferrous alloys. Water does have a number of limitations that makes it less desirable choice in certain applications. For steels with high hardenability, water quenching generally results in cooling rates higher than the critical cooling rate. It is generally admitted, as expressed by Olivier *et al.* (2004), that water quenching could lead to the development of high residual stresses leading to distortion or cracks in the component. In practice, the applicability of water as a quenching medium is restricted to plain carbon steels and some low alloy steels. The high cooling rate obtained by water also put limitations on the shape of the objects to be heat treated. Thus, only articles with simple geometric shapes can be water quenched. Water quenched objects are also prone to corrosion.

The use of oil as a quenching medium has been investigated. Although mineral oils such as SAE40 engine oil, spindle oil, and industrial lubrication oil traditionally have been commonly used as quenching media (Zhou, 1987), various vegetable oils have been used as well. Hassan and Yusuf (2005), investigated the use of various vegetable oils and found that palm kernel oil induced higher hardness values. Generally, oil quenching has a number of advantages in comparison with water. Quenching oils produces slower cooling rates as compared to water which results in the reduction of cracks or distortion in the quenched piece. Oils are used where severe quenching is not required. The cooling potential of oil can be improved by raising the temperature of the oil to between 50 – 80^{o}C (Salihu *et al*., 2013). Oils of viscosity value as low as 50 SUS (Saybolt Universal Seconds) at 40^{o}C give higher cooling rate (Rajan *et al*., 1988). A problem with oil quenchant is ageing of the bath. Immersion of very hot steel can lead to oxidation of the oil and the removal of quenched component can lead to loss of some of the liquid quenchant. Oil also has a limitation of low flash point and hence the danger of explosion and fume.

There are numerous polymers that may be dissolved in aqueous solution to produce polymer quenchants. Polymer quenchants can be formulated to provide quenching speed ranging from of fast quenching oil to that greater than water (Ester *et al*., 2013). Polymer quenchants degrade or age slowly with use and thus the quenching power depreciates, making it necessary to provide process control by periodic monitoring of the quench bath through routine maintenance (Totten *et al.*, 1997). Polymer quenchants lower the cooling rate of water by forming an insulating film on the work piece surface resulting in the effective reduction of the distortion problem (Zhoa, 1996).

Hassan *et al.*, (2011) showed that the initial temperature of the part as well as the temperature of the medium has an effect on the quenching medium. The higher the temperature of the quenching medium, the shorter will be the time for the work piece and the quenchant to attain thermal equilibrium. There is rapid loss of the cooling power of water as temperature is raised above 60^{o}C (Rajan *et al.*, 1988). Depending on the medium itself, higher bath temperatures may decrease viscosity, which affects bubble size and therefore, decrease the rate of heat transfer during the third stage of quenching. Agitation plays an important role in the effectiveness of a medium to quench a part. Agitation helps to break the insulating vapor blanket between the parts and the liquid, thereby improving or increasing the overall heat transfer rate by bringing cooler liquid into contact with the parts being quenched. Agitation forces cool liquid to be constantly circulated to the work piece in place of the hot liquid. Therefore, higher temperature differences will always exist between the medium and the surface, resulting in faster rates of heat dissipation (Mills, 1998) The increase in the quench uniformity minimizes the potential for cracking, distortion, residual stresses and un-uniform hardness around the component (Totten, 1993).

Agitation affects the hardness and the depth of hardening during quenching. Various methods have been developed to measure the effectiveness or cooling power of a quenchant, these includes magnetic test method, the hot wire test, the cooling curve test, and measuring the hardness of the quenched part. However, the cooling curve has been generally accepted as the most accurate means of determining the cooling power of a quenchant. Cooling curves are particularly sensitive to all the factors that affect the ability of quenching medium to extract heat from hot metal, these includes; quenchant type, physical properties, bath temperature and bath agitation (ASM, 1978).

MATERIALS AND METHODS

One of the experimental materials used in this study is medium carbon steel (AISI-SAE1045) with chemical composition as shown in Table 1. Chicken eggshells used for this work were collected from the local tea sellers in Hausa Market, Warri. Delta State, Nigeria. Water and Engine oil (SAE 40) were used as the quenching media.

Methods

An electric furnace with operating temperature of up to 1200°C was used. The steel samples were normalized and then austenitized at 930°C for 15 min before quenching in both the agitated and non-agitated water-based eggshell powder quenchant. Water and Engine oil (SAE 40) were used for comparative study. Preparation of the eggshell test samples involved separating the membranes from the eggshells by hand. The eggshells were boiled in hot water for 5-10 min to kill pathogens, and air dried for 2 days. The dried lump sizes were ground into powdered form with the use of a pulverizer and sieved with a mesh sieve. The particle sizes retained in 45 μm mesh size were used

X-ray diffraction analysis of the eggshell powder was carried out to determine the various phase distribution. The analysis was carried out with a Philips X-ray diffractometer. The powder X-ray diffractogram was taken using the anode material (Cobalt) at scan speed of 3°/min. From the X-ray diffraction pattern of the eggshell powder chart obtained (Figure 2) and by applying Bragg Equation (1), the inter-planar spacing of the diffracting planes in the eggshell powder was determined.

$$n\lambda = 2d \sin \theta \quad ...(1)$$

where the order of diffraction, n =1; the wavelength of the Co-Kα radiation, λ =1.79 Å; d stands for the inter-planar spacing, and 2θ, the diffraction angle. The microstructure of the surface morphology of the eggshell powder was determined using a Scanning Electron Microscope (JSM5900LV, JEOL) equipped with an Energy Dispersive X-Ray Spectroscopy. Samples were firmly held on the sample holder using a double-sided carbon tape before putting them inside the sample chamber. The SEM was operated at an accelerating voltage of 20 KV and digitized images were recorded.

The percentage of eggshell powder used ranged from 5 to 25 v/V% and were thoroughly mixed in the corresponding volume percent of water. The mixed blend was poured into five steel buckets and a1000 cm^3 of water without eggshell (100% water) was poured into another bucket to serve as the control (Table 2). In all, six quenching media were prepared, their specific latent heat of vaporization determined and the medium with the highest specific latent heat of vaporization was then chosen for the study.

The apparatus that was used in the determination of the specific latent heat of vaporization include: steam boiler, steam trap, copper calorimeter and stirrer, insulating jacket, heater, thermometer and chemical balance. The density of the eggshell powder was determined by water displacement (Archimedean Principle) method.

Table 1: The Chemical Composition of Medium Carbon Steel (AISI-SAE1045)

Elements	C	Mn	Si	Ni	Cr	Mo	Al	P	S	Ti
Wt%	0.45	0.84	0.29	0.05	0.08	<0.005	0.012	0.037	0.046	0.0025
Elements	Cu	Co	Nb	V	W	Pb	Sn	Zn	Fe	
Wt%	0.073	0.015	<0.005	0.007	<0.001	<0.005	<0.005	0.0016	97.81	

Table 2: Volume and Mass used to Determine the Specific Latent Heat of Vaporization

v/V%	5	10	15	20	25
Volume of Water Added (cm^3)	950	900	850	800	750
Volume of Eggshell Powder (cm^3)	50	100	150	200	250
Mass(g) of eggshell powder	54.5	109	163.5	218	272.5

Table 3: Specific Latent Heat of Vaporization (LV)

Quenchants	Control(water)	5v/V%	10 v/V %	15 v/V %	20 v/V %	25 v/V %
L_V(J/Kg)x10^6	2.26	2.28	2.35	2.38	2.40	2.48

The specific latent heat of vaporization, L, can be expressed by

$$L = [[(m_2 - m_1) S_2 + m_1 S_1] (\theta_2 - \theta_1)] / (m_3 - m_2) - (100 - \theta_2) S_2 \quad ...(2)$$

where

m_1= Mass of calorimeter and stirrer (kg)

m_2= Mass of calorimeter and stirrer + water (kg)

m_3= Mass of calorimeter and stirrer + water + steam (kg)

θ_1 = Initial temperature of calorimeter and contents (°C)

θ_2 =Final temperature of calorimeter + contents (°C)

S1 = Specific heat capacity of calorimeter

S_2 = Specific heat capacity of water

The temperature of steam is assumed to be 100°C.

A steel sample of 10 mm diameter and 30 mm length was employed for the purpose of plotting the cooling curve. A k-type thermocouple was fitted into a hole drilled at the geometric centre of the test piece in order to take temperature reading (Figure 1). The test piece was heated in the furnace to 930°C and quenched in water-based eggshell powder, water and engine oil (SAE 40) quenchants. The temperature and the corresponding time were recorded and the cooling

Figure 1: The Schematic Diagram of the Quench Tank

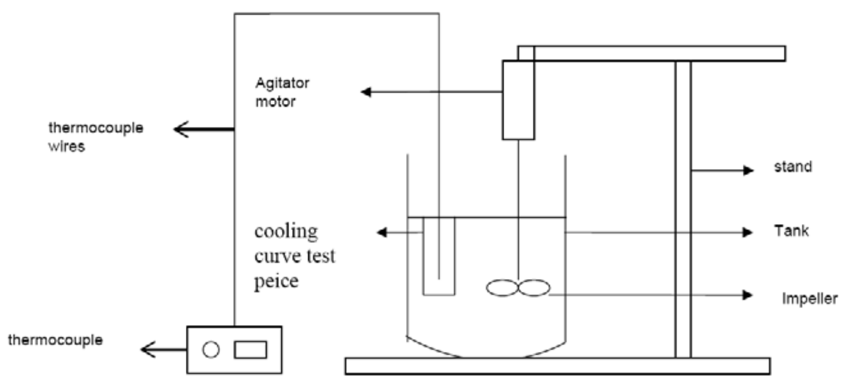
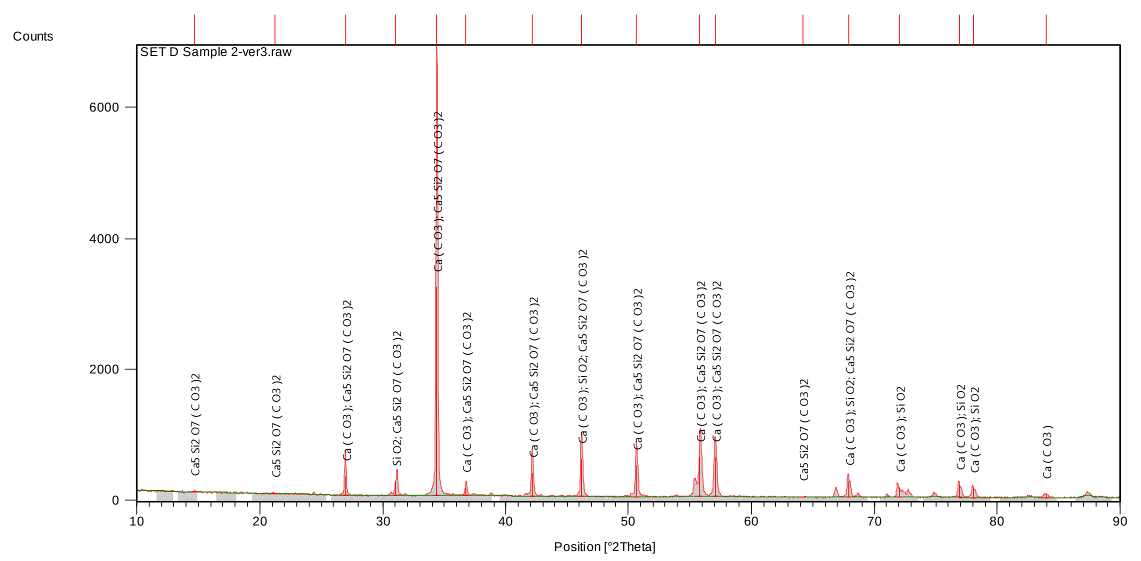


Figure 2: XRD Pattern of the Eggshell Powder Chart



curves plotted for all the quenching media. The first derivative (gradient of the tangent at a particular temperature) of the cooling curve called the cooling rate was plotted against the corresponding temperature for each quenching media. The water-based eggshell powder quenchant was agitated with an Arrow 600 variable speed impeller type agitator unit at a speed of 200 RPM, and the temperature - time data of the quenched test sample in these media was also plotted.

RESULTS AND DISCUSSION

X-Ray Diffractometer Analysis of the Eggshell Powder

The X-ray diffraction pattern of the eggshell powder obtained has many diffraction with smaller interspacing distance, the major diffraction peaks are: 26.96°, 31.06° and 72.10° with inter-planar distances: 3.84 Å, 3.34 Å and 1.52Å The phases at the peaks are: Calcite (Ca (CO_3), Quartz, syn (SiO_2) and Tilleyite (Ca_5 Si_2 O_7 $(CO_3)_2$), with scores of 82, 35 and 23 respectively (Figure 2). The complete analysis confirmed that the eggshell powder contain at least one of the elements (C, O, Si, Ca) which means that there are no harmful elements in eggshell powder. With Calcite (Ca (CO_3) having the highest score of 82, eggshell powder can be used effectively as a quenchant for steel.

Morphology of the Eggshell Powder

The microstructure of the eggshell powder reveals porous irregular shapes. The Energy Dispersive X-ray Spectroscopy (EDS) of the eggshell particles reveals that the particles contain Ca, Si, O, and C (Figure 3), with the presence of C in the carbonized eggshell particles. These elements confirm that eggshell powder consists of calcium carbonate in the form of calcite ($CaCO_3$), Quartz, syn (SiO_2) and Tilleyite (Ca_5 Si_2 O_7 $(CO_3)_2$. The analysis is in agreement with the result of the XRD.

Chemistry of Eggshell Powder in Water

When eggshell powder was mixed with water, calcium salts partially dissolved and release Ca^{2+}, HCO_3^-, CO_3^{2-} and OH^- ions through the reaction:

$$CaCO_3 + H_2O \rightleftharpoons Ca^{2+} + HCO_3^- + OH^- + CO_3^{2-} \quad ...(3)$$

Similar phenomena have been reported with calcite by Chojnacka (2005). Aside from the ions mentioned above, water-based eggshell powder quenchant also contains Na^+, Mg^{2+} and K^+ ions which originate from the eggshells. These ions may be adsorbed onto the surfaces of eggshell particles, forming a positive charge. Eggshell powder addition to water was not increased above 25% as it was observed that at higher percentages the slurry was thick and the bath could not be agitated very well.

The Specific Latent Heat of Vaporization

Density of the eggshell powder was found to be 1.09 g/cm^3 which shows that eggshell powder is very light. The specific latent heat of vaporization increased with increase in the weight percent of eggshell powder in water as shown in Table 3. The increase is due to low degree of viscosity of

Figure 3: SEM/EDS of Eggshell Powder

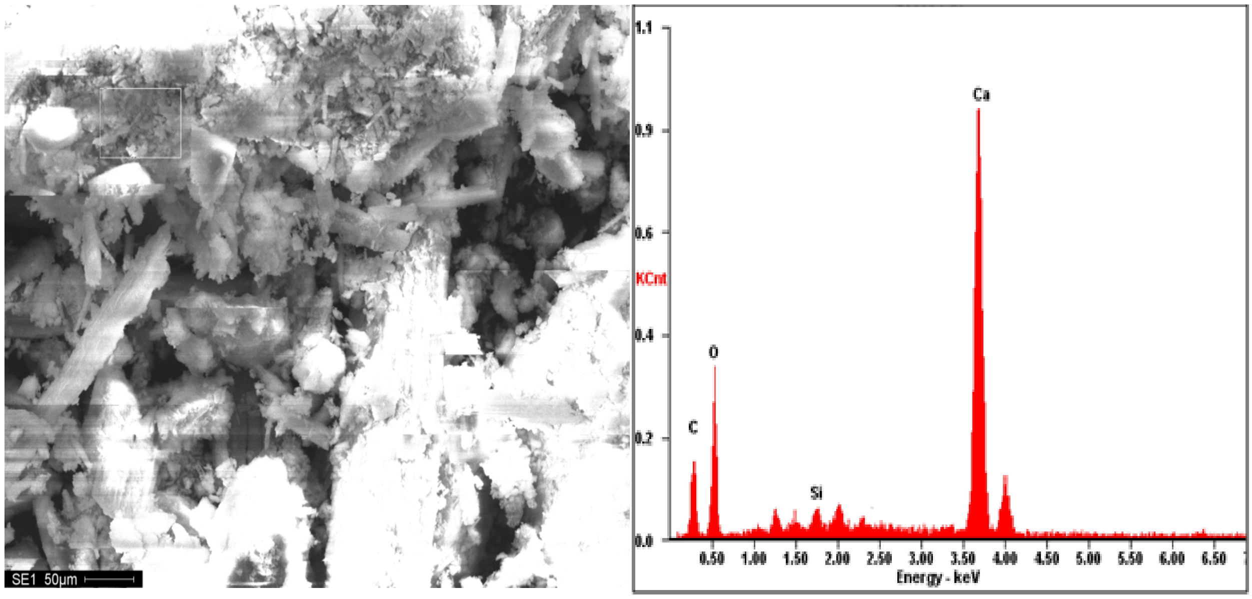


Figure 4: The Cooling Curves of the Quenching Media Investigated

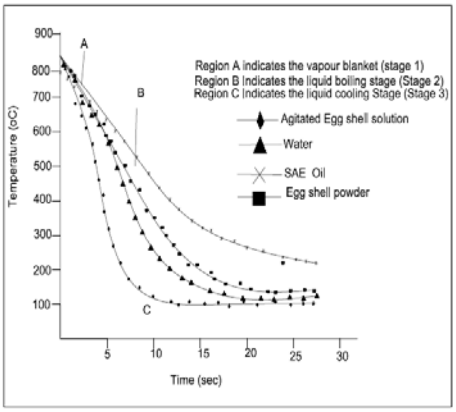
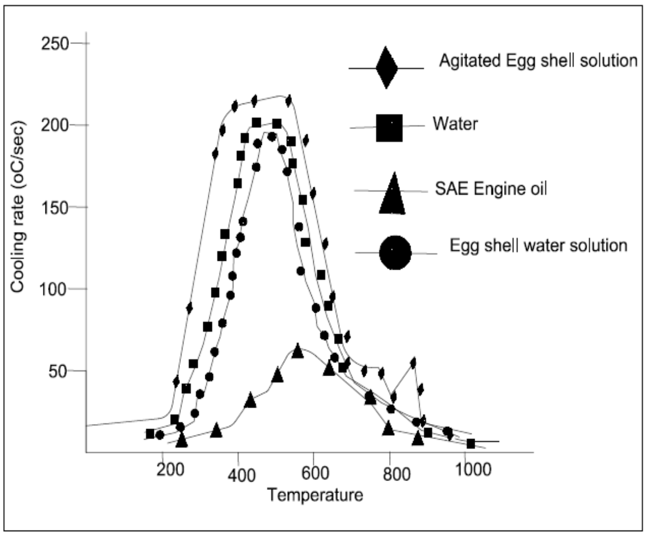


Figure 5: Graph Showing the Cooling Rate of the Quenching Media



the eggshell powder in water. As expected, the lower the viscosity, the higher the value of the specific latent heat of vaporization. However, at relatively lower viscosity, there will be easier generation of conventional currents and this seems to stabilize the cooling power at the vapour transport stage in the quenching operation.

Cooling Rate Analysis

Temperature-time curves show the heat transfer characteristic of the media. The shape of the cooling curve is representative of the various stages of the cooling mechanism during the quenching process. From the cooling curves shown in Figure 4 water-based eggshell powder quenchant (agitated) produced a very small vapour blanket stage (region A) and showed a more prolonged boiling stage (region B) as compared to water and un-agitated water-based eggshell powder quenchant. The un-agitated water-based eggshell powder quenchant gave a longer liquid boiling stage compared to engine oil (SAE 40).

From the cooling rate curve in Figure 5 agitated water-based eggshells powder quenchant gave the highest cooling rate of 225^{o}C/s at 500^{o}C. Water showed a higher cooling rate than the un-agitated water-based eggshell powder, with the highest cooling rate of 200^{o}C/sc at 315^{o}C and 173^{o}C /s at 400^{o}C respectively. The engine oil (SAE40) has the lowest cooling rate of all the quenchants investigated, with the maximum cooling rate of 52^{o}C/s at 589^{o}C.

Agitation of water-based eggshell powder quenchant increased the cooling rates of the quenchant. This conforms to the fact that agitation enhances the cooling ability of a medium (Totten *et al.*, 1993). This could be attributed to the fact that agitation increases the uniformity of the quenching media and the rate at which the heat is extracted from the samples and dissipated to the atmosphere.

CONCLUSION

The potential of water-based eggshell powder solution as a quenchant for medium carbon steel was investigated. It can be concluded from the results that 25wt% eggshell powder to water can successfully form water-based eggshell quenchant. It was observed that at a higher percentage than 25 wt% eggshell powder in water, the slurry becomes thick and the bath could not be agitated very well. Water-based eggshell powder quenchant has a higher specific latent heat of vaporization than water and agitated water-based eggshell powder quenchant gave the highest cooling rate of 225^{o}C/s at 500^{o}C when compared with water and SAE engine oil. Water-based eggshell powder quenchant can be used as a quenching medium for hardening of steel

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