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Evaluation of Water-Based Eggshells Powder as a Quenching Medium for Hardening Medium Steel

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ABSTRACT

This research work focus on the evaluation of water-based eggshells powder as a quenching medium for hardening medium steel. Medium carbon steel sample with original diameter of 12.00mm and gauge length of 50m was quenched in both the agitated and non-agitated water-based eggshell powder quenchant. The medium carbon steel samples were initially normalized and this was followed by austenitizing the samples at 930°C for 20 minutes which was eventually quench in both the agitated and non-agitated water-based eggshell powder quenchant. Hardness test was carried out on the as-quenched samples to determine the performance of the water-based egg-shell powder quenchant which was compared with water and oil. Also, the specific latent heat of vaporization, cooling rates of all the selected quenching media and metallographic analysis was carried out for each as-quenched sample in the selected media. The results obtained show that water-based eggshell powder quenchant has a higher specific latent heat of vaporization than water. The microstructure of samples quenched in agitated water-based eggshell powder quenchant revealed complete transformation of the austenite into martensite and a good degree of hardness was obtained. Therefore, water-based eggshell powder quenchant can be used as a quenching medium for hardening steel.

Keywords: Times Eggshells, Hardness, Quenching, Normalizing, Medium Carbon Steel

INTRODUCTION

Quenching is an important process in the heat treatment of steels. The important elements of any heat treatments are the controlled heating cycle and the cooling cycle. The main purpose of quenching is to induce hardness in steel part so that it will be able to perform as required when put into operation and service [1]. Its economic importance ranges from manufacturing technology in market sector such as railway, automotive, aerospace etc. Quenching is defined as the controlled extraction of heat from part by rapid cooling [2]. Quenching involves the heating of steel to temperature above the upper critical temperature, in order to convert it partially or completely to austenite. Holding it long enough to ensure the desired austenization. This is followed by rapidly cooling from the austenitizing temperature to room temperature usually in a cooling media at a rate equal to or faster than the critical cooling rate [3]. The rapid cooling results to the formation of a hard metastable micro-structure known as martensite in steel [4] and this accounts for the increase in the hardness of steel [5,6].

Quenchart is any medium that is used for extracting heat from heated steel part. The most commonly used liquid quenchants are water, oil, brine, and polymer quenchant. The main functions of a liquid quenchant is to facilitate the hardening of steel by controlling heat transfer during quenching and also to minimize the formation of undesirable thermal and transformation gradients which may lead to increase in distortion and cracking [7]. Water is the most widely used quenching medium [8]. This can be attributed to its low cost, availability, ease of handling, relatively no pollution problem, since it can be disposed easily. The layer of scale formed on the surface during heating of the work piece is broken up by water quenching, thus eliminating any further surface cleaning [9]. Water has the disadvantage of inducing crack on the quenched component due to its high cooling rate. Oil on the other hand cannot inducing enough hardness. Although, polymer quenchant can provides severity between those of water and oil but it limitation is that it varies the concentration during the quenching process and equally expensive. Brine can produce more quenching severity than water but it corrosively attacks the components and the equipment used for the quenching [10].

One of the technical challenges of quenching known is the choice of a quenching medium that will yield the desired quenched properties, such as hardness with minimum induced distortion. Therefore, there is need for the development of a quenching medium with good economics like water, but having less quenching severity and yet producing appreciable hardening. Eggshells are waste materials from hatcheries, homes and fast food industries [11,12] and can be readily collected in enormous quantities. Eggshells waste disposal contributes to environmental pollution. Challenges associated with disposal of eggshells include cost, availability of disposal sites, odours, flies and abrasiveness [9]. Nigeria produced an estimate of about 10 million tonnes of eggs annually [3]. Therefore, there is the need to take advantage of this abundance and availability of eggshell, by harnessing its economic value, and stop its wastage. This research work is aim at the evaluation of water-based eggshells powder as a quenching medium for hardening medium steel.

MATERIALS AND METHODS

Materials

The equipment used for the purpose of austenizing the medium carbon steel samples include an electric furnace with operating temperature up to 1200~C before quenching in the media. Medium carbon steel (AISI-SAE1045) was used. Table -1 shows the chemical composition of medium carbon steel (Data obtained from National Metallurgical Development Centre, Jos, Nigeria). Chicken eggshells were collected from Shaigal Farm, Amukpe, Sapele, Delta State, Nigeria. Other quenching media (Water and oil) were locally source for. Other apparatus used include: steam boiler, steam trap, copper calorimeter and stirrer, insulating jacket, heater, thermometer and chemical balance. The tensile tests were carried out on Enerpac universal materials testing machine, Rockwell hardness-testing machine (Fig. 1) was used for the hardness test, and Charpy Impact-testing machine was used for the impact test. A polishing machine, etchant and metallurgical microscope with an in-built camera were used to examine the microstructure. The photograph of the eggshell before and after grinding is shown in Figure 2.

(a.) (b)

Fig.1 (a) Digital Rockwell Hardness Machine (b) Eggshell before and after grinding Pattern

Fig. 2 Photograph of the Eggshell before and after grinding

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

Elements	C	Mn	Si	Ni	Cr	Mo	Al	P	S	Ti
Weight Percentage (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	
Weight Percentage (Wt%)	0.073	0.015	<0.005	0.007	<0.001	<0.005	<0.005	0.0016	97.81	

Table -2 Various Volume and Mass used for 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

Methods

Medium carbon steel sample with original diameter of 12.00mm and gauge length of 50m was quenched in both the agitated and non-agitated water-based eggshell powder quenchant. The medium carbon steel samples were initially normalized and this was followed by austenitizing the samples at 930°C for 20 minutes which was eventually quench in both the agitated and non-agitated water-based eggshell powder quenchant. For the purpose of comparison and performance evaluation, water and oil were used. Hardness test was carried out on the as-quenched samples to determine the performance of the water-based eggshell powder quenchant which was compared with water and oil. Also, the specific latent heat of vaporization, cooling rates of all the selected quenching media and metallographic analysis was carried out for each as-quenched sample in the selected media.

Preparation of the Eggshell Powder and the Quenching Media

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 dry 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 particles sizes that were retained in 45um mesh size were used (Fig. 2). The percentage of eggshell powder used ranged from 5 to 25v/V% and were thoroughly mixed in the corresponding volume percentage water, the mixed blend was poured into five (5) steel buckets.1000cm^3 of water without eggshell dissolution (100% water) was poured into another bucket to be used as the control. In all, six quenching media was prepared and the physical property i.e. Specific latent heat of vaporization was determined. The medium that have the higher specific latent heat of vaporization was used.

Characterization of Powdered Eggshells

The X-ray diffraction analysis of the eggshell powder was carried out to determine the various phases distribution in the samples. The analysis was carried out using Philips X-ray diffractometer. The X-ray powder diffractograms was taken using the anode material (Cobalt) at scan speed of 3o/min. From the X-ray diffraction pattern of the eggshell powder chart obtained (Fig. 3) and by applying the Bragg equation (1), the inter-planar spacing of the diffracting planes in the eggshell powder was determined using equation 1.

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

Where the order of the diffraction, n =1; the wavelength of the Co-Kα radiation, λ =1.79 Å; the inter-planar spacing, d and the diffraction angle, 2θ. The microstructure of the surface morphology of the eggshells powder was determined using a Scanning Electron Microscope (JSM5900LV, JEOL) equipped with an Energy Dispersive X-Ray Spectroscopy. The 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 20Kv. The digitized images were recorded. Table 2 shows the various Volume and Mass used for the Specific Latent Heat of Vaporization.

Determination of Specific Latent Heat of Vaporization of the Quenching Media

Eggshell powder density was determined by water displacement method. Density of the eggshell powder was calculated to be 1.09g/cm^3 and this implies that eggshell powder is a very light material.

The steps follow in determining the specific latent heat of vaporization is as follows:

- Steam boiler was filled with weight fraction of eggshell powder mixed with water (Table -2), fit into it the cork and the steam trap and boil the mixture. A copper calorimeter and stirrer of 1000g (m_1) was used.
- Calorimeter and stirrer were weighed.
- Calorimeter filled half way with water and reweigh
- Initial temperature reading taken.
- Steam was pass from steam jacket through a steam trap to remove any condensed moisture.
- Dry steam was passed into the water in the calorimeter until the temperature of the water rises above the room temperature (θ_2).
- Steam source was removed from the calorimeter and the water source is stirred continually until a steady final temperature is reached.

Final results were calculated as follow;

Mass of calorimeter and stirrer	m_1 = Kg
Mass of calorimeter and stirrer + water	m_2 = Kg
Mass of calorimeter and stirrer + water + steam	m_3 = Kg
Initial temperature of calorimeter and contents	θ_1 = °C
Final temperature of calorimeter + contents	θ_2 = °C

The procedure was repeated for other weight fractions of eggshell powder mix with water.
Let,

S_1 = Specific heat capacity of calorimeter
S_2 = Specific heat capacity of water
L = Unknown specific latent heat of vaporization of the quenchant

The temperature of steam is assumed to be 100°C. From the measurement above:

Mass of water = $m_2 – m_1$
Mass of steam = $m_3 – m_2$
Heat lost by steam condensing = $(m_3 – m_2) L$
Heat lost by condensed steam in cooling from 100°C to θ2 = $(m_3 – m_2) (100 - \theta_2) S_2$
Heat gained by water = $(m_2 –m_1) (\theta_2 - \theta_1) S_2$
Heat gained by calorimeter = $m_1S_1 (\theta_2 - \theta_1)$

Assumption;
Heat lost by steam condensing and the heat loss by condensed steam in cooling from 100°C to temperature θ_2 is equal to the heat gained by water and calorimeter.
Thus,

$$(m_3 – m_2) L + (m_3 – m_2) (100 - \theta_2) S_2 = (m_2 –m_1) (\theta_2 - \theta_1) S_2 + m_1S_1 (\theta_2 - \theta_1)$$
$$(m_3 – m_2) L + (m_3 – m_2) (100 - \theta_2) S_2 = [(m_2 –m_1) S_2 + m_1S_1] (\theta_2 - \theta_1)$$
$$(m_3 – m_2) L = (\theta_2 - \theta_1) [(m_2 –m_1) S_2 + m_1S_1] - (m_3 – m_2) (100 - \theta_2) S_2$$

Therefore;
The specific latent heat of vaporization, L, can be calculated from the expression given below (eq. 2):

$$L = \frac{[(m2 - m1)\ S2 + m1S1]\ (\theta2 - \theta1) - (100 - \theta2)\ S2}{(m3 - m2)} \quad (2)$$

Hardness Test
Hardness test was carried out using Rockwell hardness tester with 1.56mm steel ball indenter, minor load of 10kg, major load of 150kg and hardness value of 95HRC as the standard block. To ensure accuracy, the mating surface of the indenter, plunger rod and test samples were thoroughly cleaned by removing dirt, scratches and oil and calibration of the testing machine using the standard block. The test samples were placed on anvils, which act as a support for the test samples. A minor load of 10kg was applied to the sample in a controlled manner without inducing impact and then the major load of 150kg was then applied. Readings were taken when the large pointer came to rest or had slowed appreciably and dwelled for up to 3 seconds.

RESULTS AND DISCUSSION

The microstructure of the eggshell powder reveals that the size and shape of the powder vary; however, they consist of porous irregular shape powder. The Energy Dispersive X-ray Spectroscopy (EDS) of the eggshell particles reveals that the particles contain Ca, Si, O, and C, with the presence of C in the carbonized eggshell particles. The carbon presence is due to the carbonization process. These elements confirm that, the eggshell powder consists of calcium carbonate in the form of calcite ($CaCO_3$), Quartz, syn (SiO_2) and Tilleyite ($Ca_5\ Si_2\ O_7\ (CO_3)_2$. These analyses are in agreement with the result of the XRD.

Results of specific latent heat of vaporization of the quenching media showed the weight percent of eggshell powder added in water increases as the specific latent heat of vaporization increases (Table -3). The increase is as a result of low degree of viscosity of the eggshell powder in water. Usually, the lower the viscosity, the higher the value of the specific latent heat of vaporization.

From the temperature time curve which indicates the heat transfer characteristic of the media. The shape of the cooling curve (Fig. 4) is as a function of the various stages of the cooling mechanism that occurred during the quenching process.

Removal of heat from a steel component during quenching is occurs in three stages via; film boiling stage, nucleate boiling stage and convection stage. The results indicated that water-based eggshell powder quenchant (agitated) produced a very small vapour blanket stage (region A) and a more prolonged boiling stage (region B) as compared to water and unagitated water-based eggshell powder quenchant. Thus, this stage is responsible for extraction of a large amount of the heat. The cooling rate of the quenchant for medium carbon steel has effect on the microstructure and hence the mechanical properties. Figure 5 shows the microstructures of the medium carbon steel. The presence of pearlite (dark) and ferrite structure (white) support the confirmation that the steel used in the research is a medium carbon steel.

Table -3 Various Specific Latent Heat of Vaporization (L_V)

Quenchants	Control(water)	5v/V%	10 v/V %	15 v/V %	20 v/V %	25 v/V %
L_V(J/Kg)x10^6	2.27	2.29	2.38	2.40	2.43	2.49

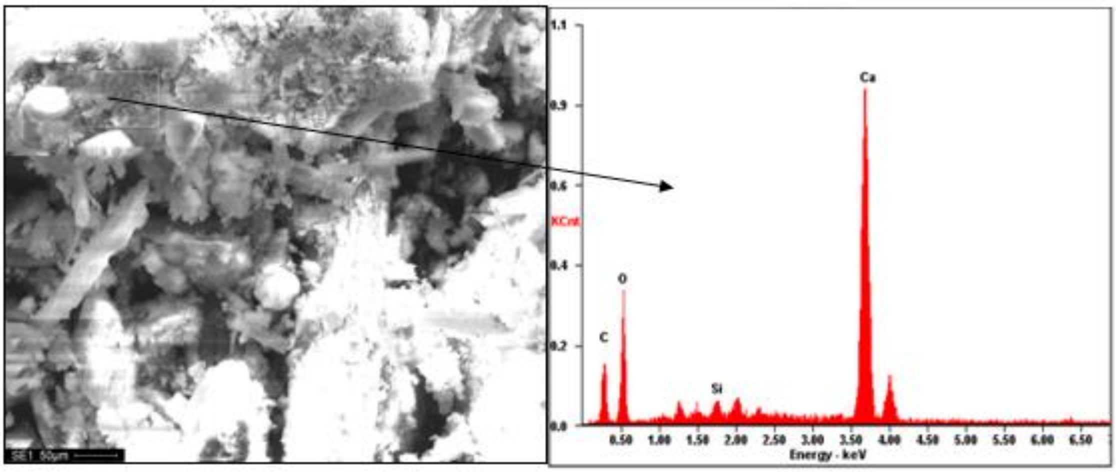

Fig. 3 SEM/EDS of Eggshell Powder

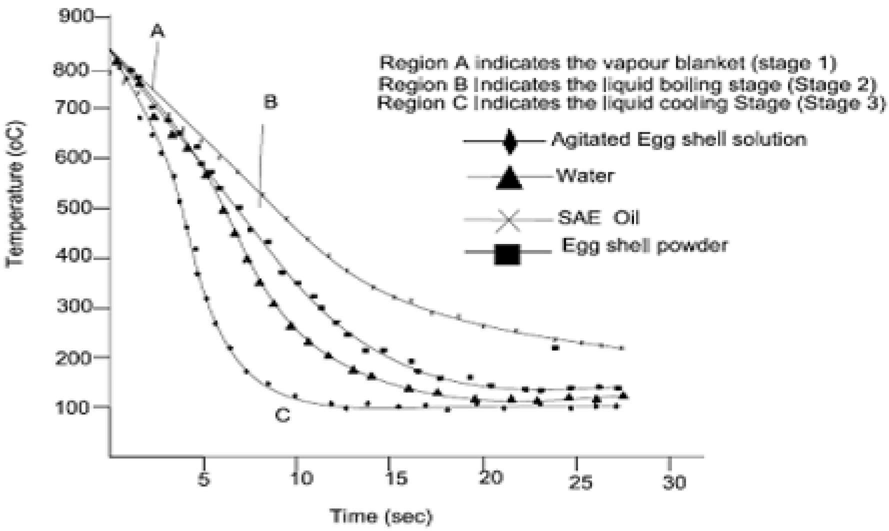

Fig.4 The Cooling Curves of the Quenching Media Investigated

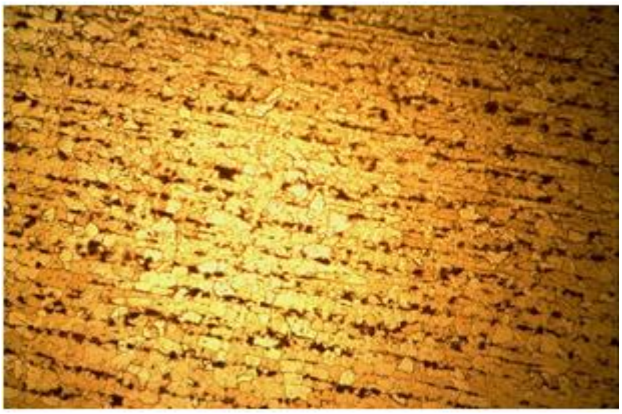

Fig.5 Cast medium carbon steel: A structure of ferrite (light) and pearlite matrix (dark) (X250)

Fig.6 Normalized Medium carbon steel: A structure of ferrite (light) pearlite matrix (dark) (X250)

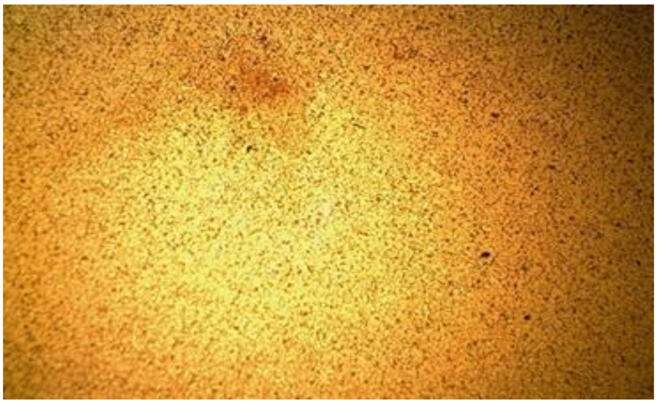

Fig.7 Full martensite structure: Medium carbon steel quenched in agitated water-based eggshells powder quenchant. (X250)

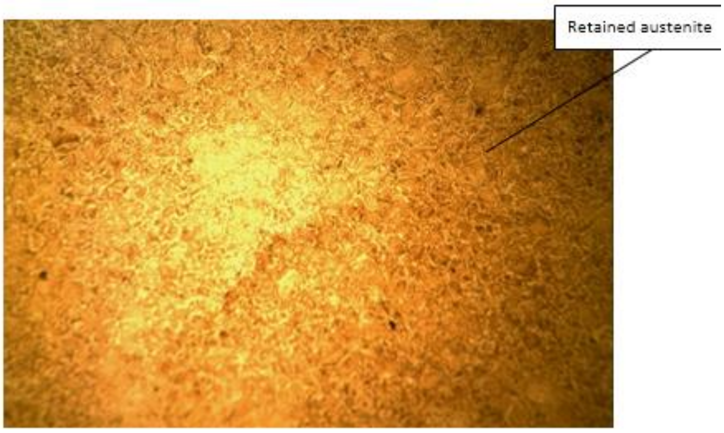

Fig.8 Martensite structure with retained austenite: Medium carbon steel quenched in unagitated water-based eggshells powder quenchant. (X250)

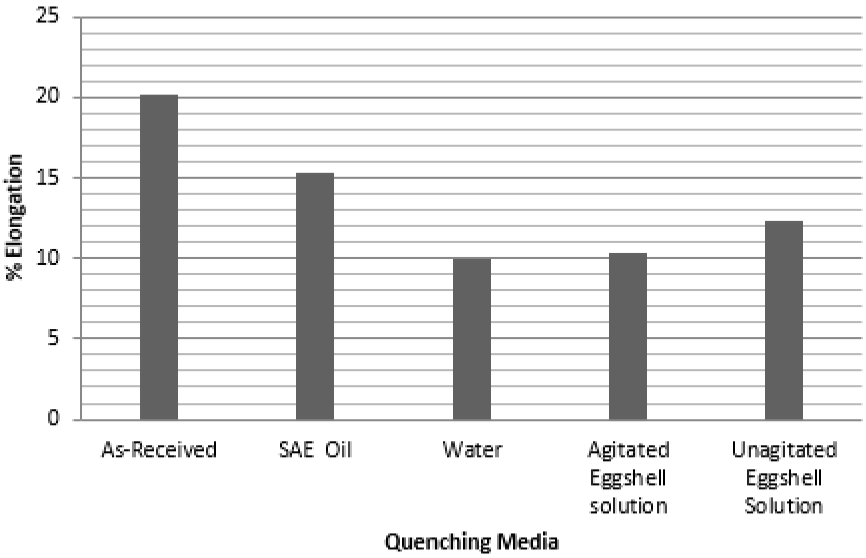

Fig. 9 % Elongation of plain carbon steel in as-received and quenched conditions

Table -4 Summary of Developed Microstructure of Plain Carbon Steels in Various Conditions

Heat Treatment	Microstructure
Originally	Pearlite in ferrite matrix
Normalization	A highly pearlitic matrix
Quenching with water	Martensite with partial austenite retained
Un-agitated water-base eggshell powder as quenchant	Martensite present with austenite retained=
Quenching with Oil	Martensite present, austenite retained and slight increase in bainite
Agitated water-base eggshell powder quenchant	Formation of fully Martensite structure

Table -5 Summary of Hardness Test

Quenched Samples	Hardness (HRC)
Original	48.35
Oil	50.01
Water	59.34
Agitated eggshell quenchant	62.34
Unagitated eggshell quenchants	52.09

The Normalized structure revealed a structure of grain boundary ferrite and pearlite (Fig. 6). For the as-quenched samples in water-based eggshell powder quenchant (agitated), it was observed that the microstructure of the medium carbon steels shows the full formation of martensite structure (Fig. 7) while the sample that was quenched in water-based eggshell powder without agitation did not shown full martensite. However, the microstructure revealed that there is some retained austenite in the martensite (Fig. 8). Table -4 shows the summary of developed Microstructure of plain carbon steels in various conditions. The summary of hardness test is shown in Table 5. The results show Agitated eggshell quenchant has the highest degree of hardness.

The hardening process by quenching in all the liquid media reduced the impact strength and percentage elongation of the medium steel. The as-received sample gave the highest impact strength value, and the samples quenched in water gave the least impact strength (Fig. 9). Samples quenched in the unagitated water-based eggshell quenchant and oil (SAE40) produced higher impact strength. The lower impact energy of water could be attributed to the transformation and thermal stress after water quenched. These results are in line with the microstructure observed for water, oil (SAE 40) and water-based eggshell quenchant Water-based eggshell quenchant quenched produced toughness in between that of oil (SAE 40) and water.

CONCLUSION

This research work focus on the evaluation of water-based eggshells powder as a quenching medium for hardening medium steel. The results obtained showed that water-based eggshell powder quenchant has a higher specific latent heat of vaporization than water. The microstructure of samples quenched in agitated water-based eggshell powder quenchant revealed complete transformation of the austenite into martensite and a good degree of hardness was obtained. Therefore, water-based eggshell powder quenchant can be used as a quenching medium for hardening steel.

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