

HARDNESS AND MICROSTRUCTURE OF 0.60%C STEEL HARDENED IN TRANSESTERIFIED NEEM OIL

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ABSTRACT

The hardness, impact strength and microstructure of 0.60%C plain carbon steel quenched-hardened in transesterified neem oil (TN) are reported in the study. Fresh neem oil (FN) was transesterified using methanol. Afterwards, steel samples normalized and then austenitized at 850°C for 40 minutes and then quenched in TN, FN and SAE40. The quenchant used as bench mark was SAE40. The as-quenched samples' hardness and impact strength tested. Additionally, microstructural analysis on the as-quenched samples was carried out. TN-quenched sample exhibits higher hardness and impact strength as compared to FN-quenched parts. In all the quenched samples, martensite and retained austenite were observed. The investigation shows that TN gives good combination of hardness and impact strength. Therefore, TN is recommended to be used as quench medium for 0.60%C (AISI 1060) steel.

Keywords: Transesterification, neem oil, FTIR, AISI 1060 steel, hardness, impact strength.

INTRODUCTION

Cracks that occur during water quenching have been a problem to the metallurgical industries especially with high carbon steel (Olson, 2001). Any high carbon steel that cracks because of quench hardening rendered scrap and this eventually leads to economical losses. It has been established that mineral oil (such as SAE40) is a suitable quenchant for high carbon and low alloy steels as it provides moderate and uniform cooling rate (Hassan *et al.*, 2010; Joseph *et al.*, 2015). Nonetheless, recently heat treatment industries have shifted focus to the use of vegetable oil, due to increasing cost of mineral oil and its several environmental and disposal liabilities (Dodo, 2015). On the other hand, the use of vegetable oils is partially restricted due to their high thermal oxidative instabilities which lead to low heat extraction ability (Ramesh and Prabhu, 2014). Notwithstanding, there are lots of methods for improving these drawbacks, such as the chemical modification of vegetable oils; the genetic modification of fatty acids; the direct addition of antioxidants and viscosity index modifiers (MacNutt and He, 2016). Among these methods, the first reported to be the most interesting for improving thermal oxidative stability. According to Turco *et al.* (2017), chemical modifications mainly involve altering the acyl (C=O) and alkoxy (O-R) functional groups and unsaturations of the triglyceride molecules of the vegetable oil and fats. Esterification and transesterification reactions are commonly used to modify the acyl group by forming new esters with better physicochemical properties (Madankar *et al.*, 2013). The conversion of vegetable oils to methyl esters (transesterified oil) for quench application has been reported in literatures, including: (Otero *et al.*, 2014; Dodo *et al.*, 2019). However, despite the abundance of the neem oil in India, South East Asia and West Africa, the applicability of transesterified neem oil for quench application has not been reported. Hence, the focus of this study is the assessment of quenching performance of transesterified neem oil using 0.60%C plain steel.

MATERIALS AND METHODS

Modifications Process

Neem oil was chemically modified through transesterification of the esterified FN. The oil esterified to reduce the FFA (free fatty acid) content to a value less than 0.5%. The variables of the transesterification process as reported by Malgwi and Encinar *et al.* (2010) for maximum conversion of triglyceride to methyl esters adopted. Mass of the methanol and catalyst (NaOH) that was used in the mixture was 21.7 % and 1% respectively. The calculated mass of the NaOH was dissolved in the measured methanol and the mixture poured into the measured quantity of the neem oil (100g). Afterwards, the solution was heated to 60°C on magnetic stirrer and agitated for 1hour. In the end, methyl esters was separated from the glycerol by pouring the solution into separating funnel. The denser glycerol drained after draining the transesterified oil.

Heat Treatment Operations

Spheroidize annealing was conducted purposely to improve machinability of the 0.60%C steel used. Samples were heated to 850°C and soaked for four hours for the transformation of cementite lamella to spheroids; after which the samples furnace-cooled. Steel samples heated to 850°C and soaked for 60 minutes for the necessary transformation to occur and the attainment of homogenization after which air-cooled. This normalizing heat treatment was conducted purposely to remove the effects of undesirable structures due to machining. In the Hardening treatment, samples austenitized at 850°C, soaked for 40 minutes and then quickly quenched in the TN, FN and SAE40. All the quench media maintained at room temperature of 27°C. The samples grouped under the condition of as-received (ASR), normalized (NLD) and quenched.

Mechanical Tests

Hardness Measurement

The samples used for the metallography were then subjected to hardness test using the Rockwell hardness indentec universal

hardness testing machine (scale C), model-8187.5 LKV (B) with diamond cone (120°) indenter. Each sample mounted on the machine with the polished surface faced up, three indentations made on the surface, and the depth of indentation made was measured by the electronic scale which converts the depth measurement to the corresponding hardness value. The average of the three hardness value determined and recorded.

Izod Impact

The method employed is in line with ASTM E23. Before the test, the pendulum was set to a potential energy position of 162.75 J. The sample with standard dimension gripped vertically in a vice, the trigger was released and the pointer showed the energy absorbed in breaking the sample. Subsequently, Impact strength calculated. The same repeated for other test pieces.

Microstructural Examinations

All the samples both as received and heat-treated prepared for OM and SEM. The samples ground on grit papers of 180, 240, 320, 400, 600 and 800 sizes with water as lubricant. Polishing was carried out by spraying 1µm Alumina paste on the disc of universal polishing machine. Samples then etched in aqua regia solution using the immersion method, immersed for 50 seconds. Finally, etched samples snapped with OM and SEM.

Fourier Transform infrared Spectroscopy

IR spectra of the FN and TN recorded on a Fourier Transform infrared spectroscopy. The frequency and intensity of the band obtained automatically by using the find peak command of the instrument software.

RESULTS AND DISCUSSION

Hardness and Impact strength

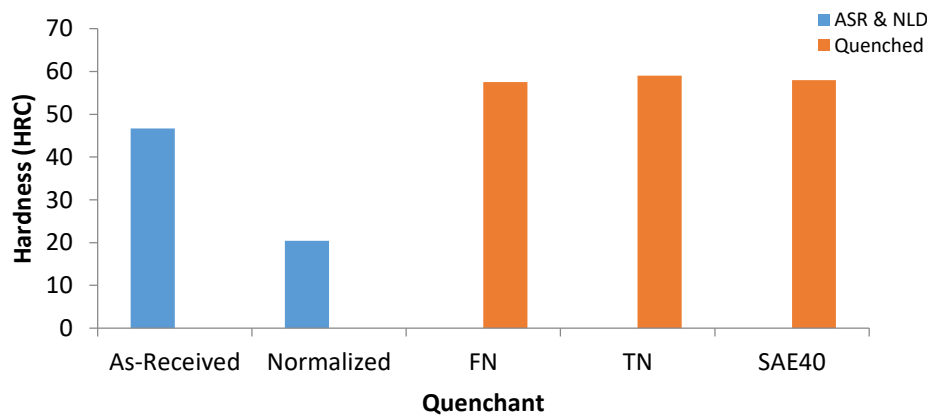


Fig. 1: Hardness values of as received, normalized and quenched samples

In Fig. 1, highest hardness value of 59 HRC was attained by the sample quenched in TN. Even though, hardness value of FN-quenched sample is mildly lower than that of the TN-quenched sample, TN exhibits excellence quenching performance as no sludge formed after quenching. Accordingly, TN can be used frequently without deterioration

of the quenching performance unlike FN. The improved performance is due to the transesterification treatment done on FN. In a similar instance, Dodo *et al.* (2019) identified transesterified mahoganyseed oil as having higher quenching performance in comparison to the fresh oil.

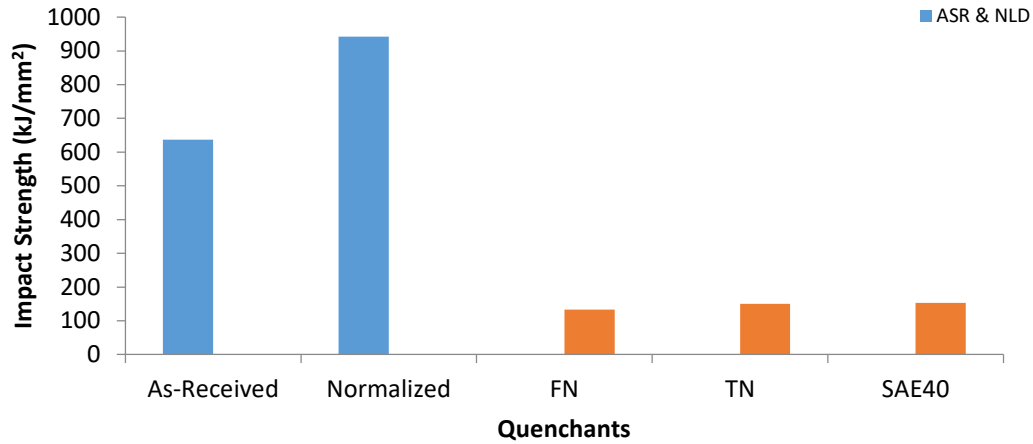


Fig. 2: Impact strength of as received, normalized and quenched samples

From Fig. 2, it is evident that sample quenched in FN exhibited least impact energy of 133 kJ per mm². Also, impact strength of 636 and 942 kJ/mm² noted from the as received and normalized samples respectively. Compared to samples quenched in neem oils, sample quenched in SAE40 indicated

highest impact strength. Moderate impact strength of 150 kJ/mm² is displayed by the TN-quenched sample. Thus, TN showed better heat extraction ability due to enhanced thermal stability as a result of the transesterification. However, Alabi *et al.* (2012) reported that water quenched steel at bath temperature of 95 °C exhibits higher impact strength.

Microstructures

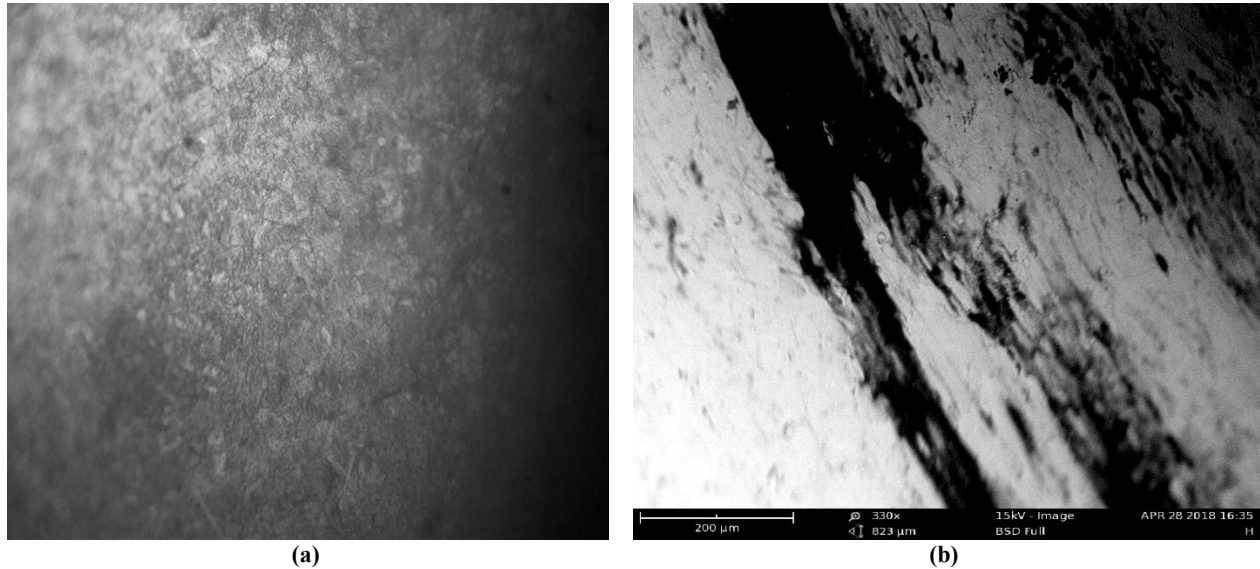


Fig. 3: (a) Optical micrograph (x200) & (b) SEM Image of the 0.60%C steel quenched in FN; Aqua regia etch

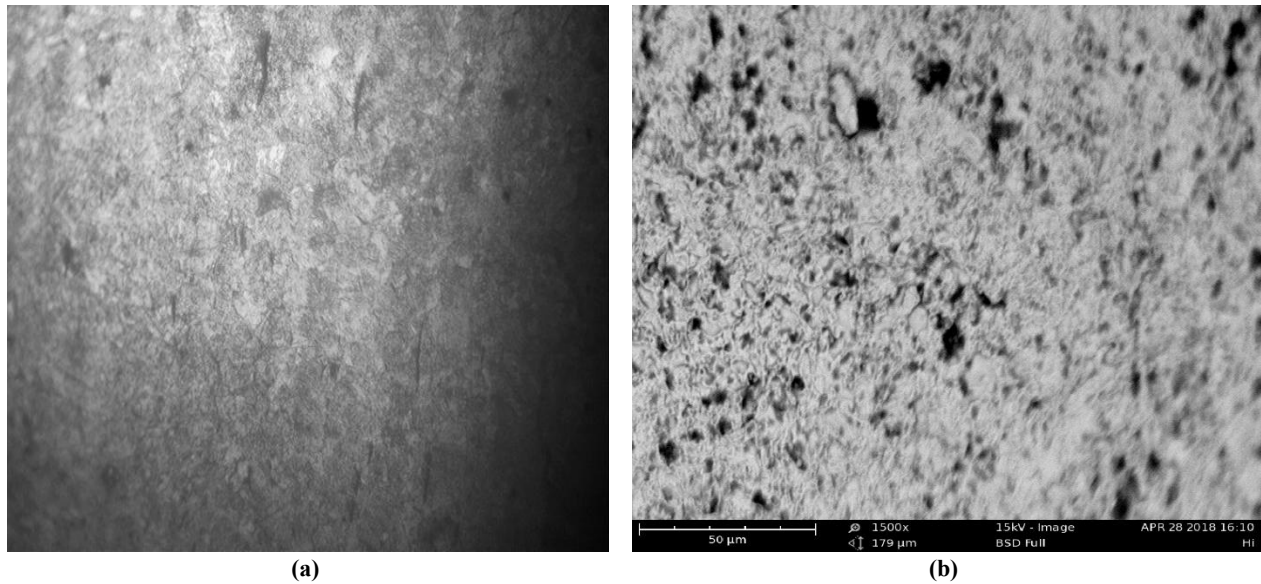


Fig. 4: (a) Optical micrograph (x200) & (b) SEM Image of the 0.60%C steel quenched in TN; Aqua regia etch

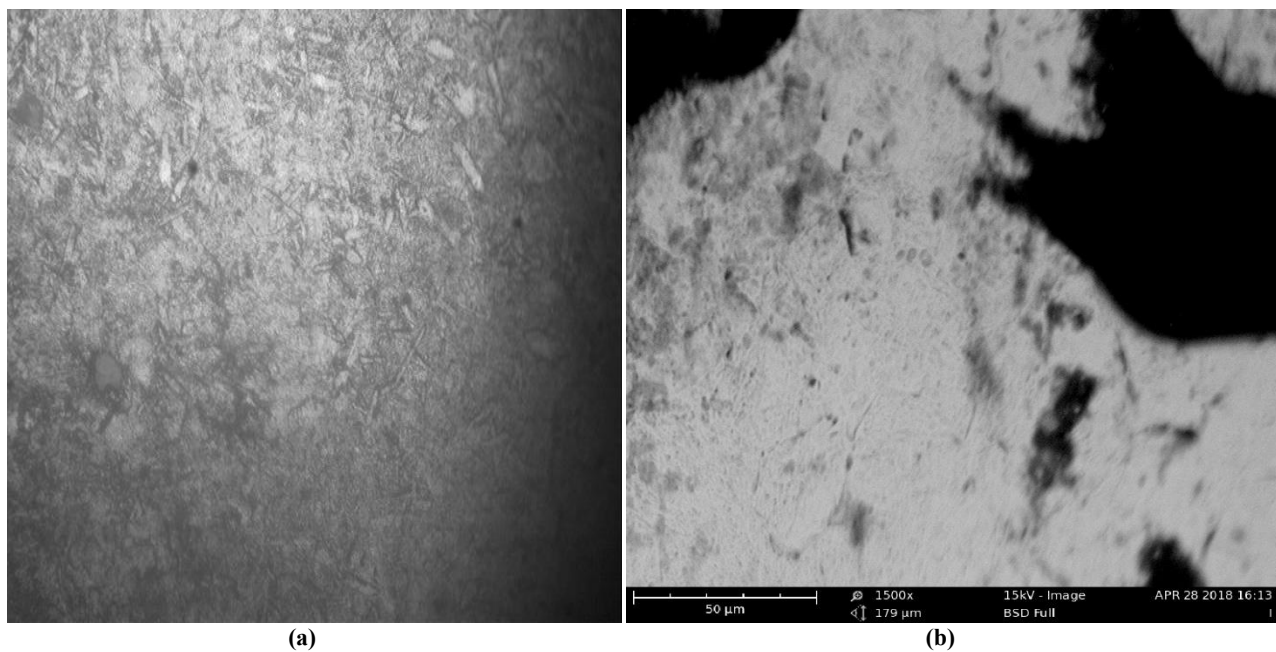


Fig. 5: (a) Optical micrograph (x200) & (b) SEM Image of the 0.60%C steel quenched in SAE40; Aqua regia etch

In Figs. 3-5, the structures consist of lath and plate of ferrite saturated with carbon which is much alike martensite (Rao and Sudheer, 2016). This indicates the structures reveal martensite with small amount of retained austenite. TN-quenched sample proved to have higher hardness (Fig. 1) due to higher content

of martensite compared to other quenched samples. The superior cooling characteristics resulted from the removal of β -hydrogen atoms of glycerol molecule upon transesterification (Hamizah and Jumat, 2014).

Fourier Transform infrared Spectroscopy

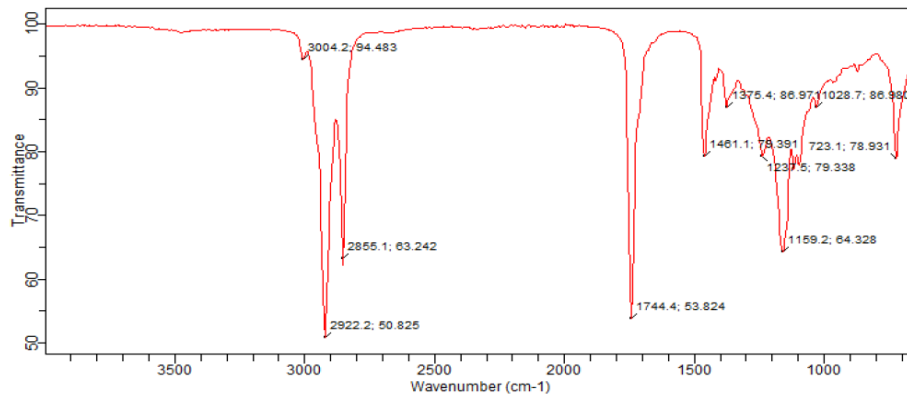


Fig. 6: FT-IR spectrum of FN

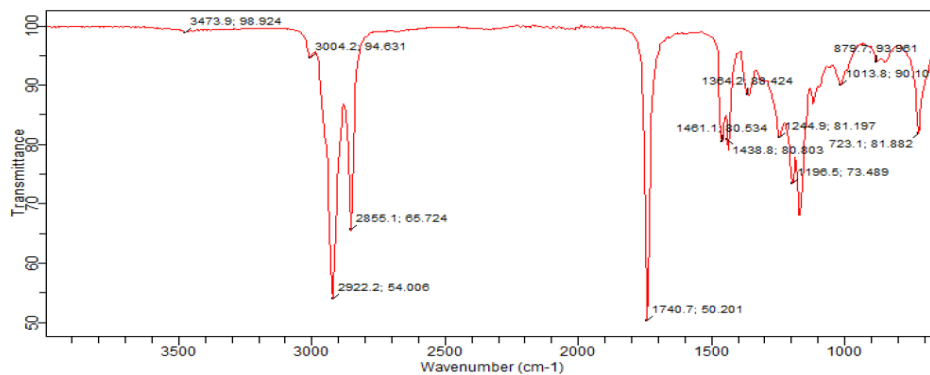


Fig. 7: FT-IR spectrum of TN

The FT-IR analysis for the fresh and transesterified neem oils portrayed many absorption peaks as showed in Figs. 6 - 7. In the Figs. the respective overtone stretching vibration of C=O for the esters and vibrational stretching of C-H in C=C-H in the fatty acids present in oils are observed by peaks at 3474 cm^{-1} and 3004.2 cm^{-1} . Similarly, the CH₃- methyl ester group in the TN is shown at wavenumber 1438.8 cm^{-1} . Siatis *et al.*, (2006) as well made alike observation.

CONCLUSIONS

The effect of transesterified neem oil as a quench medium for 0.60%C steel has been explored using hardness values, impact strength and microstructure. Based on the results, AISI 1060 steel can be quenched effectively using TN. Similarly, FN caused formation of martensite structure in the AISI 1060 steel. However, sludge formed limited its repeated use. Further, TN-quenched steel parts possess higher hardness as compared to FN and SAE40-quenched parts. Additionally, due to the enhanced thermal stability TN can be used several times. Therefore, TN could be recommended as a substantial replacement for mineral oil in hardening of AISI 1060 steel.

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