

DOI: 10.5604/01.3001.0015.0255

Volume 108 Issue 2 April 2021 Pages 68-74 International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

Experimental study of the mechanical and corrosion properties of ethyl silicate resin applied on low carbon steel

M. Ali a,*, J.H. Mohmmed b, A.A. Zainulabdeen b

^a Power Mechanics, Al-Furat Al-Awsat Technical University, Babylon Technical Institute, Iraq

^b Materials Engineering Department, University of Technology, Iraq

* Corresponding e-mail address: inb.mus12@atu.edu.iq

ORCID identifier: 10 https://orcid.org/0000-0003-3785-6104 (M.A.);

b https://orcid.org/0000-0002-2608-9176 (J.H.M.)

ABSTRACT

Purpose: This work aimed at evaluating the properties of the ethyl silicate-based coating that can be applied on low carbon steel.

Design/methodology/approach: Two mixture ratio types (2:1, and 3:2) of resin and hardener respectively were used to prepared two specimen models (A and B).

Findings: It found that some mechanical properties (tensile, hardness, and impact strength) of ethyl silicate resin were evaluated according to standard criteria.

Research limitations/implications: The effect of heat treatments at various temperatures (100, 150, and 200°C) and holding at different times (10, 20 & 30) min on hardness was investigated.

Practical implications: Moreover, an open circuit potential corrosion test with a solution of 3.5% Sodium Chloride at room temperature and 60°C was used to determine the corrosion resistance of low carbon steel specimens coated with the two mixture types.

Originality/value: The effects of mixture ratios (for resin and hardener) and heat treatment conditions on properties of ethyl silicate-based coating were studied. From obtained results, acceptable values of tensile, hardness, and toughness were recorded. Increasing heat treatment temperature and holding time leads to enhance hardness for both model types. An open circuit potential (OCP) tests show that there is an enhancement of protective properties of ethyl silicate coatings with mixture type B in comparison with type A was achieved. Generally, the results indicate that specimen model B has higher properties as compared with specimen model A.

Keywords: Ethyl silicate resin, Polymer coating, Low carbon steel

Reference to this paper should be given in the following way:

M. Ali, J.H. Mohmmed, A.A. Zainulabdeen, Experimental study of the mechanical and corrosion properties of ethyl silicate resin applied on low carbon steel, Archives of Materials Science and Engineering 108/2 (2021) 68-74. DOI: https://doi.org/10.5604/01.3001.0015.0255

PROPERTIES

1. Introduction

Surface engineering is a multidisciplinary activity intended to improve the properties and features of the surface of engineering parts so that their function and serviceability can be enhanced [1]. The coating is one of the most important methods utilized for the modification of the surface properties and protection of the components from corrosion and aggressive conditions. The metal corrosion protection efficiency is dependent on many variables like quality of coating, metal characteristics, metal-coating interface properties, and the aggression of the environment [2]. Undoubtedly, ferrous alloys are one of the most important alloys used in the different industries [3]. But on of its limitation is the low corrosion resistance, i.e. to return to its origin, and become useless, this result in losses amounting to about 10% of the world output [4]. So, the enhancement of corrosion resistance of ferrous, i.e. to keep the ferrous alloys in their usable form, and extend the life or improve the performance of steel components working in severe conditions have often an important matter. This can be attained by developing new material, applying the coating, or a combination of these [5]. various coatings have been developed for protecting ferrous alloys against corrosion in modern technologies. Many coating systems ranging from polymeric or metallic to ceramic (oxide base), out of which ethyl silicate polymeric based coatings have additional advantages of shock-resistance, lightweight, and excellent chemical properties comparing to nowadays coatings.

Ethyl silicate, often referred to as tetraethoxysilane or tetra ethylortho silicate (TEOS), $Si(OC_2H_5)_4$, is a tetra functional silicon alkoxide (Fig. 1) synthesized in two ways; firstly, via reacting ethanol with silicon tetrachloride (SiCl₄) and secondly, via reacting ethanol with silicon metal [6].



Fig. 1. Chemical structure of tetra ethyl ortho silicate (TEOS) [6]

Many plastics, like acrylic resin, and polycarbonate resin, have downside that scratches quickly occur on the surface, greatly detracting from the quality of items. However, by utilizing products that use ethyl silicate, a very thin film of SiO_2 is created on the surfaces, that resulting in enhances the resistance of abrasion and reduces scratching.

Moreover, heat and weather resistance is dramatically enhanced, by using ethyl silicate onto a variety of different substrates, and the fields of application of the substrate are widely extended. Additionally, the light transmission can be boosted and avoid the reflection of the light more operatively by adding it to plastic or glass. Moreover, the particles' surfaces can be covered by a SiO₂ membrane, thus enhancing the dispersibility of the particles and other properties.

In addition, ethyl silicate was applicable in the world, especially when the liquid precursor of silica (SiO₂) is demanded. [7]. Silica fine particles are produced by ethyl silicate which acts as a binder adherence ceramic to refractories [8]. Corrosion resistance coating was applied when it combined with zinc [9]. It can be used for the consolidation of wall paintings [10,11]. One of its uses as an electrical insulator to the surface of semiconductor chips after vaporization and thermal decomposition of ethyl silicate to make silica gel. This technique is used to manufacture integrated circuits [12]. There is continuous development in ethyl silicate chemical applications include silicon cross-linking and versatile chemicals [12]. Ethyl silicate as a polymer or monomer form has wide applications in silicon-based materials as liquid precursors [13]. Silica colloids as binder agents were produced by hydrolysing and condensing ethyl silicate, to use in manufacturing ceramic materials with a high resistance to corrosion and superior mechanical strength and dielectric properties. However, although the ethyl silicate-based coatings were studied by many researchers, there are few works that treat the effects of mixture ratios (for resin and hardener) and heat treatment conditions on properties of ethyl silicate-based coating. Researchers have studied the effect of the mechanical properties improvement that increased with the increase of the chromium ratio in the four cases in general to reduce the rate of the corrosion [14]. The powder metallurgy method was used in order to prepare the produced pad friction materials to test the hardness and roughness [15]. Hence, to compensate for the lack in this issue, the current study is aimed at the evaluate of some mechanical properties of the ethyl silicate resin with different mixture ratios and under different heat treatment conditions. Additionally, the suitability of this coating to protect the steel sheet against corrosion was investigated.

2. Materials and specimens

2.1. Materials

A commercial ethyl silicate (ETS) based coating with its hardener (Hardener 1, type 008 7380 0360), were used in the current study. Two mixing methods (2:1 and 3:2) for resin and hardener respectively were prepared and used in tests. Specimens of low carbon steel sheets $(10 \times 10 \times 0.8)$ mm with the chemical composition shown in Table 1 were used as a substrate in the current work.

2.2. Mixtures and specimens preparation

A manual method was used to mixing ethyl silicate with its hardener. Two mixing types (2 risen - 1 hardener and the 3 resin -2 hardener) were used to prepare two specimens model symbolled (A and B) respectively. To perform the tensile test, a mixture of two series was poured into a mould in form of a tensile specimen according to ASTM D638 (Fig. 2) and curing for about 24 hours. Also, specimens were cut to perform impact strength and hardness tests. Moreover, for polishing the low carbon steel specimens, emery papers with have various grades ranging from 80 to 1000 were employed. After that, the specimens were washed and cleaned with water and ethanol to be prepared for the coating process. Lastly, the low carbon steel samples recovered with the two mixture models of ethyl silicate coatings as shown in Figure 3 to prepare it for corrosion testing (OCP test). Furthermore, samples for the hardness test were cured and thermally treated (Fig. 4) in an oven at different times and temperatures. Heat treatment conditions and sample designation were illustrated in Table 2.





Fig. 2. Tensile test specimen: a) scheme, b) before test, c) after test



Fig. 3. Coated low carbon steel sample



Fig. 4. Heat treated samples

Specimen type	Symbol	Heat treatment conditions		
	A	As received		
	A ₁₁	Heated to 100°C then hold to 10 minute		
	A ₁₂	Heated to 150°C then hold to 10 minute		
	A ₁₃	Heated to 200°C then hold to 10 minute		
٨	A ₂₁	Heated to 100°C then hold to 20 minute		
A	A ₂₂	Heated to 150°C then hold to 20 minute		
	A ₂₃	Heated to 200°C then hold to 20 minute		
	A ₃₁	Heated to 100°C then hold to 30 minute		
	A ₃₂	Heated to 150°C then hold to 30 minute		
	A ₃₃	Heated to 200°C then hold to 30 minute		
	B∘	As received		
	B ₁₁	Heated to 100°C then hold to 10 minute		
	B ₁₂	Heated to 150°C then hold to 10 minute		
	B ₁₃	Heated to 200°C then hold to 10 minute		
D	B_{21}	Heated to 100°C then hold to 20 minute		
D	B ₂₂	Heated to 150°C then hold to 20 minute		
	B ₂₃	Heated to 200°C then hold to 20 minute		
	B ₃₁	Heated to 100°C then hold to 30 minute		
	B ₃₂	Heated to 150°C then hold to 30 minute		
	B33	Heated to 200°C then hold to 30 minute		

 Table 2.

 Heat treatment conditions and samples classification

3. Methods

3.1. Mechanical tests

The Izod impact toughness test according to ASTM D256 standard was used in this work Figure 5. Five individual readings were performed and averaged to get an acceptable value of the impact strength of a material. The total energy of the impact test affected by the notch shape, size, and length of the test specimen.



Fig. 5. Schematic of Izod test specimen

The hardness measurements of the specimens were carried out by using a shore tester type D according to ASTM D 2240. The hardness was determined by taking five readings on each specimen and averaging them.

3.2. Open circuit potential measurement

To evaluate corrosion resistance, the open circuit potential (OCP), as well called the free corrosion potential, was used (Fig. 6). For each specimen, open circuit potential



Fig. 6. The electrochemical cell and open circuit potential (OCP) test setup

(OCP) was carried out for a half-hour. To get this value, the specimens were placed in a 3- electrode cell (coated steel specimen as a working electrode (WE), platinum wire service as counter electrode (CE), and a saturated calomel electrode (SCE) used as reference electrode) immersed in 3.5% sodium chloride (NaCl) solution with ensuring connections establishment in the potentiostat set. The OCP value is the criterion employed to make a comparison between the dissolution of specimens in the same environments within the solution.

4. Results and discussion

The results of the tensile test for two types of specimens were evaluated by using stress – strain diagrams presented in Figure 7. The result reveals that the tensile stress of specimen B much higher than that of specimen A. Meantime, the elongation for specimen B dramatically decreasing as compared with specimen A. the main reason for this behaviour is the increase in hardener content in specimen B. The increase in the mixture ratio in specimen B maybe leads to fill the more open porosities embedded in ethyl silicate structural and consequently increases the structure integrity and strengthen it.

On other hand, Table 3 displays the results of the hardness test. In general, it noticed the hardness values were enhanced with increasing both the temperature and time holding of heat treatment. At the same time, it is observed the hardness properties of specimen B are better as compared with specimen A. This may be attributed to that heat treatment leads to enhance the formation of higherordered structures of samples and consequently enhancing the hardness properties.



Fig. 7. Stress-strain diagram of two specimens

		Results Holding time				
Specimens	Temperature, °C					
		10 min	20 min	30 min		
	100	60	65	67		
А	150	65	63	66		
	200	69	70	72		
	100	72	71	75		
В	150	77	78	77		
	200	77	78	78		

Table 3. Hardness test results

The results of toughness properties of impacted specimens indicate that there are slight differences between the two types of specimens. The toughness value for specimen A was 27 while for specimen B was 26. This difference may be associated with a slight increase in brittleness in specimen B as a result of increasing the hardener. At the same time, by referring to the strain-strain diagrams of the two samples, A and B, the areas under the two curves are somewhat close, which gives the same indication that the toughness value of the two samples is approximately corresponding. This suggests that the modification in the mixing ratio has no significant inverse impact on the toughness property of the resultant coating.

Table 4.Open circuit potential results

Model	OCP (V) in 3.5 wt.% NaCl solution	OCP (V) in 3.5 wt.% NaCl solution (60°C)
base	-53	-57
Sample A	-44	-49
Sample B	-43	-48

The Open-Circuit Potential (OCP) corrosion test results are presented in Table 4. It is clear that the dissolution of samples B less than of that samples A, also the samples immersed in 3.5 wt.% NaCl solution at static conditions and room temperatures have fewer OCP values comparing to those exposed with the same condition but at high temperature. This means that the immersed samples in 3.5 wt.% NaCl solution at room temperature nobler and have a tendency to dissolve were lower. The most important point on the tests performed at both of room and high temperature was that the samples had approximately the same opencircuit potential. It can be concluded from this test that at these two temperatures, coatings behaviours with different mixture ratios were approximately the same. Actually, these values were indicative of more active OCP values of the specimens at elevated temperatures. On other hand, the results reveal that the resistance to corrosion of coated samples significantly improved for both temperature conditions. This suggests that ethyl silicate is powerful and efficient method to protect steel from a corrosion. This chemical inertness is attributed to the chemical inorganicorganic formulation of the ethyl silicate resin and its small monomer sizes. The same trend was observed by [16,17] for ethyl silicate-coated steel penal by using electrochemical method and nanocomposite modified ethyl silicate.

5. Conclusions

In current work, some properties of ethyl silicate-based coating were evaluated. The effect of two mixture ratio types (A and B) of resin and hardener on mechanical properties (tensile, hardness, and impact) and corrosion resistance were studied. Moreover, the effect of heat treatments conditions (temperature and holding time) on hardness were investigated. It can be summarized from the experimental finding the following conclusions:

- 1. Generally, sample B has properties higher than the ones of sample A.
- 2. Hardness of heat-treated samples was improved by increasing the temperature and holding time of the heat treatment process.
- 3. Ethyl silicate is a powerful and efficient method to protect steel from corrosion.
- 4. The immersed samples in 3.5wt.% NaCl solution were nobler at room temperature, and less dissolve comparing to those at high temperature.

References

- J.H. Mohmmed, A.H. Ataiwi, I.A. Mahmood, Factors Affecting the Glass-Ceramic Coating: Composition, Heat Treatment, and Mill Addition, Lap Lambert Academic Publishing, Moldova, 2012.
- [2] G. Parashar, D. Srivastava, P. Kumar, Ethyl silicate binders for high performance coatings, Progress in Organic Coatings 42/1-2 (2001) 1-14. DOI: https://doi.org/10.1016/S0300-9440(01)00128-X
- [3] A.A. Zainulabdeen, N.Y. Mahmood, J.H. Mohmmed, The effect of polymeric quenching media on mechanical properties of medium carbon steel, IOP Conference Series: Materials Science and Engineering 454/1 (2018) 012053.

DOI: https://doi.org/10.1088/1757-899X/454/1/012053

- [4] A.H. Ataiwi, I.A. Mahmood, J.H. Mohmmed, Studying and modeling the effects of quartz addition and heat treatment on corrosion properties of ceramic coating, Engineering and Technology Journal 29/12 (2011) 2564-2579.
- [5] J.H. Mohmmed, A new high thermal shock resistant onelayer glass-ceramic coating for industrial and engineering applications, Journal of Achievements in Materials and Manufacturing Engineering 82/2 (2017) 70-76. DOI: <u>https://doi.org/10.5604/01.3001.0010.2357</u>
- [6] B.H. Dolph, An Evaluation of Ethyl Silicate-Based Grouts for Weathered Silicate Stones, PhD Thesis, UCLA, 2014.

[7] M. Narisawa, H. Ukon, H. Mabuchi, K. Okamura, Y. Kurachi, SiC microsphere derived from phenolic resin—Ethyl silicate heterogeneous precursors with carbothermic reduction, Journal of Applied Polymer Science 94/4 (2004) 1612-1618. DOI: <u>https://doi.org/10.1002/app.21082</u>

[8] H. Sarydikmen, N. Kushkonmaz, Microstructure and phase analysis of ethyl silicate-bonded alumina-zircon refractories, Refractories and Industrial Ceramics 46/3 (2005) 212-216. DOI: <u>https://doi.org/10.1007/s11148-005-0088-0</u>

[9] F. Hoshyargar, S. Ali Sherafati, M.M. Hashemi, A new study on binder performance and formulation modification of anti-corrosive primer based on ethyl silicate resin, Progress in Organic Coatings 65/3 (2009) 410-413. DOI: <u>https://doi.org/10.1016/j.porgcoat.2009.02.006</u>

[10] N. Laura, S. Duchêne, V. Vergès-Belmin, C. Dandrel, D. Giovannacci, W. Nowik, Comparative in Situ study of nanolime, ethyl silicate and acrylic resin for consolidation of wall paintings with high water and salt contents at the chapter hall of Chartres cathedral, International Journal of Architectural Heritage 14/7 (2020) 1120-1133. DOI: https://doi.org/10.1080/15583058.2020.1731628

- [11] J.S. Pozo-Antonio, D. Noya, C. Montojo, Aesthetic effects on granite of adding nanoparticle TiO₂ to Si-based consolidants (ethyl silicate or nano-sized silica), Coatings 10/3 (2020) 215. DOI: https://doi.org/10.3390/coatings10030215
 - DOI: https://doi.org/10.3390/coatings10030215
- [12] O. Alhussein, E. Elgorashi, H. Bayahia, A Novel Method for Synthesis of Ethylsilicates and its

Applications, Asian Journal of Chemistry 28/12 (2016) 2741-2744.

DOI: https://doi.org/10.14233/ajchem.2016.20106

- [13] D.C. Bradley, R.C. Mehrotra, I.P. Rothwell, A. Singh, Alkoxo and Aryloxo Derivatives of Metals, Academic Press, London, 2001.
- M. Ali, I. Alshalal, F.N. Al Zubaidi, A.R. Yousif, Improvement of Corrosion and Erosion Resistance Properties for Cast Iron, IOP Conference Series: Materials Science and Engineering 881/1 (2020) 012068. DOI: <u>https://doi.org/10.1088/1757-899X/881/1/012068</u>
- [15] M. Ali, I. Alshalal, A.A. Abtan, A.R. Yousif, J.H. Mohmmed, Effect of Nano-Sized SiO₂ Particles Addition on The Surface Roughness and Micro Hardness of Copper-Based Friction Materials, Journal of Mechanical Engineering Research and Developments 44/2 (2021) 104-111.
- [16] E. Akbarinezhad, H.R. Faridi, A. Ghanbarzadeh, Evaluation of zinc rich ethyl silicate primer by applying electrochemical methods, Surface Engineering 25/2 (2009) 163-166.

DOI: https://doi.org/10.1179/026708408X375280

[17] E. Akbarinezhad, M. Ebrahimi, F. Sharif, M.M. Attar, H.R. Faridi, Synthesis and evaluating corrosion protection effects of emeraldine base PAni/clay nanocomposite as a barrier pigment in zinc-rich ethyl silicate primer, Progress in Organic Coatings 70/1 (2011) 39-44.

DOI: https://doi.org/10.1016/j.porgcoat.2010.09.016



© 2021 by the authors. Licensee International OCSCO World Press, Gliwice, Poland. This paper is an open access paper distributed under the terms and conditions of the Creative Commons Attribution-NonCommercial-NoDerivatives 4.0 International (CC BY-NC-ND 4.0) license (https://creativecommons.org/licenses/by-nc-nd/4.0/deed.en).