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Abstract

Purpose – This study aims to apply novel anticorrosive pigments containing silica fume-phosphates (Si-Ph), which were prepared using core-shell technique by covering 80-90 per cent silica fume (core) with 10-20 per cent phosphates (shell) previously, to play dual functions simultaneously as anticorrosive pigments in coating formulations and as an anticorrosive admixture in concrete even if it is not present in the concrete itself. Two comparisons were held out to show the results of coatings on rebars containing core-shell pigments in concrete, and concrete admixtured with silica fume can perform a dual function as anticorrosive pigment and concrete admixture. The evaluation of corrosion protection efficiency of coatings containing core-shell pigments and those containing phosphates was performed.

Design/methodology/approach – Simple chemical techniques were used to prepare core-shell pigments, and their characterization was carried out in a previous work. These pigments were incorporated in solvent-based paint formulations based on epoxy resin. Different electrochemical techniques such as open-circuit potential and electrochemical impedance spectroscopy were used to evaluate the anticorrosive efficiency of the new pigments.

Findings – The electrochemical measurements showed that concrete containing coated rebars with core-shell pigments exhibited almost similar results to that of concrete admixtured with silica fume. Also, the anticorrosive performance of coatings containing Si-Ph pigments offered protection efficiency almost similar to that of phosphates, proving that these new pigments can perform both roles as anticorrosive pigment and concrete admixture.

Originality/value – Although the new Si-Ph pigments contain more than 80 per cent waste material, its performance can be compared to original phosphate pigments in the reinforced concrete.

Keywords Coatings, Pigments, Surface coatings, Concrete, Silica fume, Waste, Corrosion, Inorganic compounds, Electrochemistry, Anticorrosive pigments, Core-shell

Paper type Research paper

Introduction

It is well known that reinforced concrete corrosion can be found on the priority list of concrete durability considerations (Koch *et al.*, 2002). Concrete rebars are naturally protected from corrosion by passivation that occurs at the steel/concrete interface inside the alkaline cementitious matrix. However, this passivation can be lowered either by a decrease in the pH value ($pH < 9$) due to carbonation or by the presence of chloride ions that start an expansive corrosion products on the reinforced steel, which cause external pressure, leading to cracking and damaging the surrounding concrete (Binici *et al.*, 2012). Concrete structures such as bridges, buildings and marine constructions may suffer severe impairment due to the consequence of reinforced steel corrosion (Page, 1998).

The corrosion control methods for the concrete reinforced steel can differ from cathodic protection, surface coating of the rebars (e.g. organic coating), coating on the concrete surface or application of mineral admixtures such as silica fume. Adding silica fume as mineral admixture has numerous targets, including the amendment of bond strength between concrete and rebars, impermeability, corrosion protection and workability (Jalili *et al.*, 2009).

Silica fume is a waste by-product of ferrosilicon alloys industry. It is highly reactive supplementary cementitious material which has a small size with amorphous concentrated SiO_2 particles. Since a while, silica fume has been used as

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anticorrosive admixture in concrete due to its small particle size and high pozzolanic activity, which can help in filling the pores between concrete particles, leaving no other ways for the corrosive materials to approach the surface of the rebars (Siddique, 2011; Wild *et al.*, 1995; Ahmed and Abdel-Fatah, 2012).

Although there is high importance in adding such materials as admixture in concrete for rebars corrosion protection, organic coatings are still considered as the most effective, economic and widely used methods in corrosion control that can be easily applied and maintained because of its efficient protection of metal substrates from corrosion through various mechanisms (Jašková and Kalendová, 2012). Pigments are one of the main components of coating formulations; part of their function is to positively affect the physical and chemical properties of the coating and its corrosion inhibition activity.

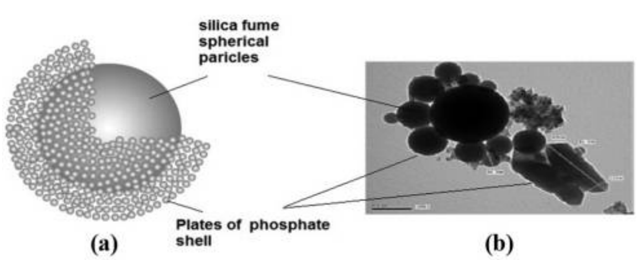
Phosphate pigments are counted as leading anticorrosive pigments, and zinc phosphate derivatives were the first suggested as safe replacements to the toxic chromate pigments in paint formulations (Buxbaum and Pfaff, 2005; Deyá *et al.*, 2010). Phosphate-based coatings are one of the most famous coatings for the corrosion protection of concrete rebars due to their high corrosion protection accomplishments in different media and pHs.

In accordance with the high evolution in the area of coatings and pigments, new techniques were raised to produce advanced materials that can play the pigments role; among these methods is the "core-shell" technique, which was used in this work to prepare silica fume-phosphates (Si-Ph) pigments. The reason of coating on the core particle was different issues such as surface modification, increasing functionality, stability, dispersibility and control release of core (Ahmed, 2009; Ahmed and Mohamed, 2010). The prepared Si-Ph core-shell pigments have been characterized in previous studies (Ahmed *et al.*, 2015; Ahmed *et al.*, 2016) (Figure 1).

In this paper, these new pigments were introduced as coating component to play double roles:
- as anticorrosive pigments containing both phosphates and silica fume; and
- the role of anticorrosive admixture in concrete.

The work will focus on:
- proving that the role of Si-Ph pigments even if they are present as rebar coating can be extended to give the concrete the same effect as admixture concrete with silica fume; and
- comparing the performance of the new Si-Ph and phosphate pigments to state the difference in their efficiency and whether they can be considered as efficient alternatives for phosphates.

Figure 1 Structure of previously prepared core-shell pigments



Notes: (a) Schematic of core-shell structure; (b) TEM micrograph of core-shell Structure
Sources: Ahmed *et al*. (2015); Ahmed *et al*. (2016)

Experimental work

Materials

- The cement used in this study is CEM I type ordinary Portland cement; its chemical analysis is given in Table I.
- The used silica fume in preparing pigments and in the concrete preparation was obtained from the Egyptian Ferrosilicon factory in Aswan, Egypt.
- The used reinforcement steel is deformed ribbed steel with 12 mm diameter, and its chemical composition is given in Table II.

Methods

Reinforced bars preparation

Deformed ribbed steel rebars with 12 mm diameter were prepared using emery papers to remove materials such as mill scales, grease, rust, oil and dirt. The steel rebars were then flushed with xylene and left to dry.

Preparation of coating formulations

Three paint formulations were prepared based on *pigment/binder (P/B)* ratio of 2:12 by *using epoxy resin (Bisphenol A type with polyamide hardener)* and 40 per cent loading of tested pigments from the total solids. The formulations contain:
- silica fume-zinc, strontium and zinc strontium phosphates core-shell pigments;
- zinc, strontium and zinc strontium phosphates; and
- one formulation containing silica fume.

Table I Chemical composition of ordinary Portland cement

Ordinary Portland cement (OPC)	Concentration of main constituents (Wt.%)
CaO	66.15
SiO_2	17.84
Al_2O_3	4.17
Fe_2O_3	4.40
SO_3	3.85
TiO_2	0.56
MgO	1.11
Na_2O	0.74
K_2O	0.30
P_2O_5	0.17
ZnO	0.23
CuO	0.01
NiO	0.01
MnO	0.13

Table II Chemical composition of reinforced steel

Element	C	Mn	P	Si	Cu	Fe
Wt.%	0.30	0.61	0.040	0.20	0.19	Balance

The paints were prepared using ball mill and then were applied by brush on the prepared rebars surface to have a uniform film thickness. These films were allowed to be cured for three days in a laboratory environment (temperature = 23 ± 2°C, and relative humidity = 40 ± 5 per cent) (Table III).

Measurement of coatings thickness
Dry film thickness of all coatings was measured using magnetic thickness gauge. All the coated rebars were adjusted to have dry film thickness ranging from 100 to 150 μm.

Mechanical properties of paint films for different coatings

The mechanical properties are indications of the flexibility, elasticity and strength of the paint film. These tests provide a basis for studying the mechanism of protective organic coatings action. The mechanical properties of coating systems were evaluated according to following standards:

- The surface hardness of the paints was measured using pendulum apparatus (ASTM D 6577-00, 2007).
- Impact resistance (ASTM D2794) test revealed the height of the free fall of a weight (1,000 g) at which the paint film still resists damage.
- The test of the resistance of the coating against cupping in Erichsen cupping tester (ASTM D 5638-00, 2007) was performed to identify the resistance of the paint film against ongoing deformation of a coated steel panel with a pressed-in 20 mm steel ball. The result of the test gives cupping (mm) after the first disturbance of the coating.

Concrete preparation and curing
Two concrete mixes were prepared and cured according *to ASTM C 192/C 192M-06,* and the used mix design is given in Table IV.

Electrochemical measurements
Concrete cubes with diameter of 5 × 5 × 5 cm with embedded coated rebars were used in the corrosion tests. Two electrochemical methods were selected for the assessment of the corrosion resistance of the different coatings. These methods are given below:

- Open-circuit potential (OCP).

The target of this test is to estimate the corrosion endurance of coated steel rebars in concrete by measuring the half-cell potential. The concrete specimens were totally immersed in 3.5 per cent NaCl solution, and the steel rebar (working electrode) was connected to the positive terminal of a voltammeter while the negative terminal was connected to Ag/AgCl electrode (reference electrode) placed adjacent to the specimen. This circuit was established for every single sample separately:

- Electrochemical impedance spectroscopy (EIS).

The measurements were carried out on concrete cubes involving the steel rebars coated with paint formulations containing core-shell pigments as working electrode. The reference electrode was $Hg/Hg_2Cl_2/Cl^-$ of E = 240 mV versus reference hydrogen electrode, and the auxiliary electrode was a platinum sheet. The used electrolyte was 3.5 per cent NaCl solution, and EIS measurements were carried out by enforcing 10 mV sinusoidal potential through a frequency domain from 35 kHz down to 100 mHz. All the experiments were executed in an aerated solution at room temperature.

The effect of different coatings on the concrete mechanical properties

- Bond strength (pull out test).

This test is executed to define the effectiveness of the coating materials on the adherence between steel rebar and concrete; a concrete cylinder of 600 mm height and 150 mm diameter containing an integrated steel bar at its center was tested by a hydrodynamic equipment, in which the mounted concrete was fixed while the outer part of the rebar was connected to the driving holder. This driving holder was then moved, and the ultimate force required to pull the rebar off was recorded according to *ASTM A944-10.*

Table III Paint formulations

Formulation / Pigments (Wt.%)	Formulation containing silica fume	Formulation containing core-shell pigments	Formulation containing phosphate pigments
TiO_2		9.8	
Talc		10	
Fe_2O_3		10	
$BaSO_4$		11	
Silica fume (Si)	27.2	–	–
Core-shell pigments (Si-Ph)	–	27.2	–
Phosphates (Zn, Sr, Zn.SrPh)	–	–	27.2
Total pigments		68	
Total binder		32	
Pigment/Binder ratio (P/B)		2.21	
Epoxy:hardner ratio		5:3	

Table IV Concrete mix design

Ingredient	Mix 1	Mix 2 (admixture with 10% silica fume from cement per cent)
Cement (kg/m^3)	350	315
20 mm aggregate (kg/m^3)	620	830
10 mm aggregate (kg/m^3)	580	420
Sand (kg/m^3)	660	590
Silica fume	–	35
Water/cement ratio (Total water of hydration)	0.60	0.71
Slump (mm)	70	77

Results and discussion

Mechanical properties of coatings

The mechanical properties involving hardness, ductility and impact resistance of paint formulations having phosphates and core-shell pigments were studied to compare the effect of the different pigments on the paint film.

Hardness

As shown in Figure 2, the hardness results proved that coatings containing core-shell pigments offered almost the same hardness as those containing phosphates, and it was noticed that better results of coatings containing silica fume-zinc-strontium phosphates (Si-Zn.SrPh) pigments were obtained.

Ductility

In Figure 3, it can be stated that paints containing core-shell pigments showed slight increase in ductility results than those including phosphates.

Impact resistance

The impact resistance results represented in Figure 4 announced that all the coatings containing core-shell and phosphate pigments were alike, except those containing Si-Zn.SrPh mixed pigment, which showed better results than that of phosphates.

Figure 2 Hardness of paint films containing phosphates and core-shell pigments

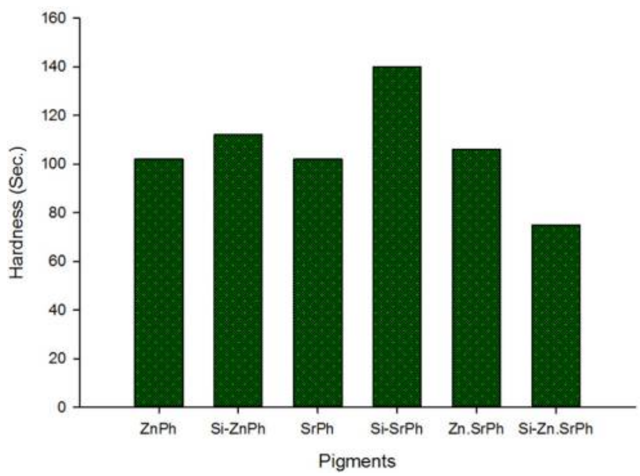


Figure 3 Ductility of paint films containing phosphates and core-shell pigments

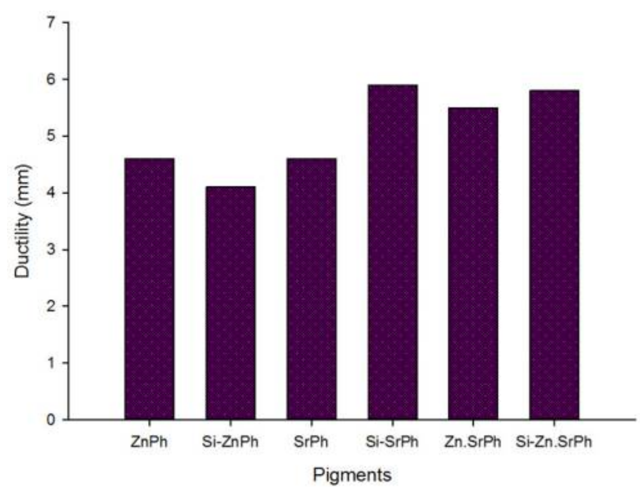
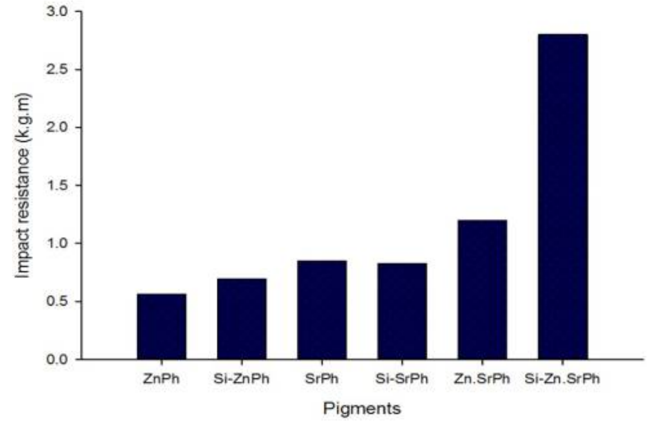


Figure 4 Impact resistance of paint films containing phosphates and core-shell pigments



In general, it can be deduced that paint films containing prepared core-shell and phosphate pigments exhibited almost similar results in the mechanical tests of the different coatings. These results manifested that using core-shell pigments that are based mainly on the silica fume in their structure affected positively the mechanical properties of the films, especially when compared to those containing phosphates. It is also clear that the presence of silica fume (core) in the paint film with high percentage compensated the defect that can show up due to the presence of low concentration of phosphates that are in the shell (only 10-20 per cent). The effect of silica fume on the mechanical properties of coatings can be announced as its spherical particles can assist the plates of phosphates found in the paint film. In addition, these spherical particles can move freely through the paint film, providing a tactile effect giving better mechanical properties, and that was clear in coatings containing Si-ZnPh, which showed better results in hardness and impact resistance tests (Azadi *et al.*, 2011; Yajun and Cahyadi, 2003).

Corrosion resistance evaluation

Case I

The first comparison was held out compare and evaluate the performance of coatings containing core-shell pigments in concrete and concrete admixtured with silica fume. This was done through the following methods.

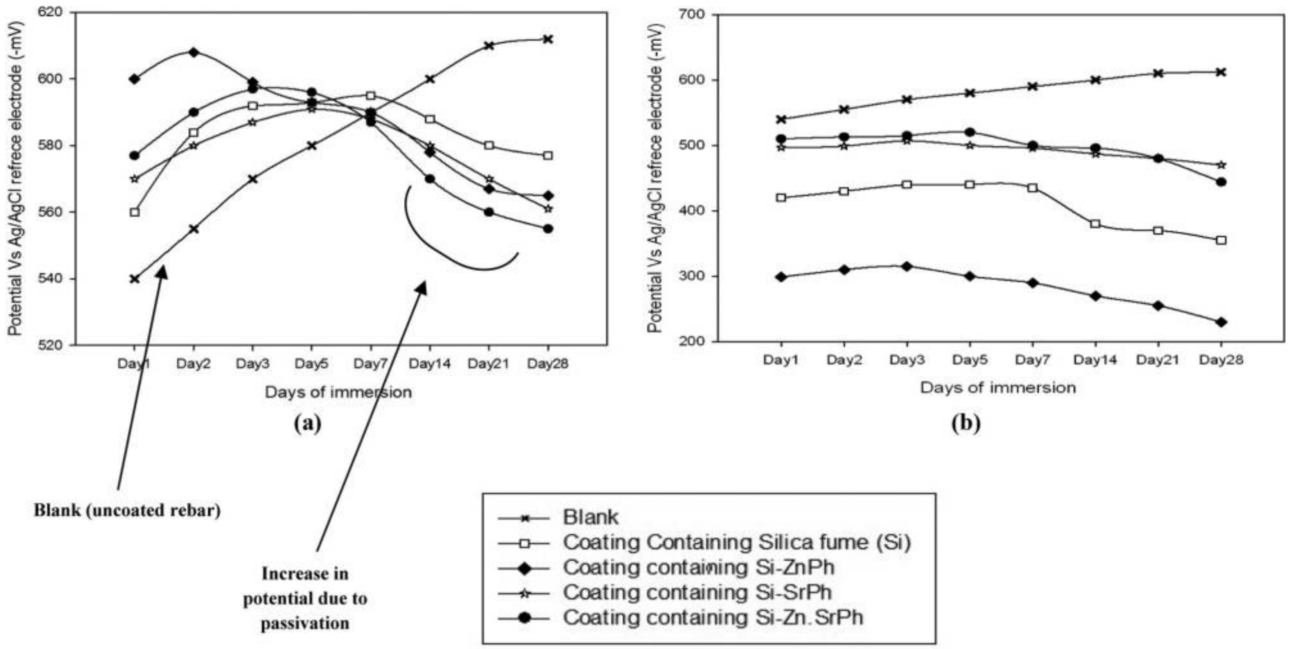
Open-circuit potential

This test is performed to indicate the corrosion protection behavior, and the obtained results can be confirmed using EIS.

It is obvious from Figure 5(a) and (b) that coatings containing the core-shell pigments showed similar potential behavior in both of concrete and concrete mixed with silica fume. This was clear in the drop of potential that was detected in the first week, and then the potential started to have slight fluctuation.

In case of (uncoated) rebars, a potential decrease was detected along the immersion time, indicating the increase in corrosion reaction at the rebar surface, which can be dedicated to the depassivation and the high attack of corrosive materials. The rebars found in the concrete mixed with silica fume showed a slight potential increase than other samples,

Figure 5 Open-circuit potential for coatings containing silica fume and core-shell pigments in concrete



Notes: (a) Rebars in normal concrete; (b) rebars in concrete admixtured with 10 per cent silica fume

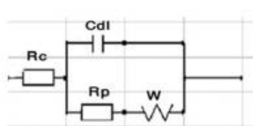
indicating an increase in the passivation process. The results of the two cases are still close to each other, revealing that the presence of silica fume in the coatings can strongly play role as an admixture with concrete even it is only present in coating films on rebars. EIS can help in giving quantitative results of the coated rebars resistance in the different types of concrete, confirming the results obtained from the open-circuit potential measurements.

Electrochemical impedance spectroscopy

The impedance spectra for different Nyquist plots were analyzed by fitting the experimental data to simple equivalent circuit, which is illustrated in Figure 6. This circuit includes Warburg diffusion element (W), the medium resistance (Rc) and the double-layer capacitance (Cdl), which is placed parallel to the coating resistance (charge transfer resistance) Rp.

Figures 7 and 8 and Tables V and VI show the Nyquist plots and the polarization resistance (Rp) of coated rebars through immersion time. From the given data, it is clear that the uncoated specimen displayed the least corrosion resistance (Rp), while coated rebars in both types of concrete showed close Rp values with the advantage of the concrete admixtured with silica fume, which exhibited slightly higher resistance results, confirming the results obtained from the open-circuit measurements. It is also clear that coatings containing Si-ZnPh

Figure 6 Equivalent circuit for fitting EIS data



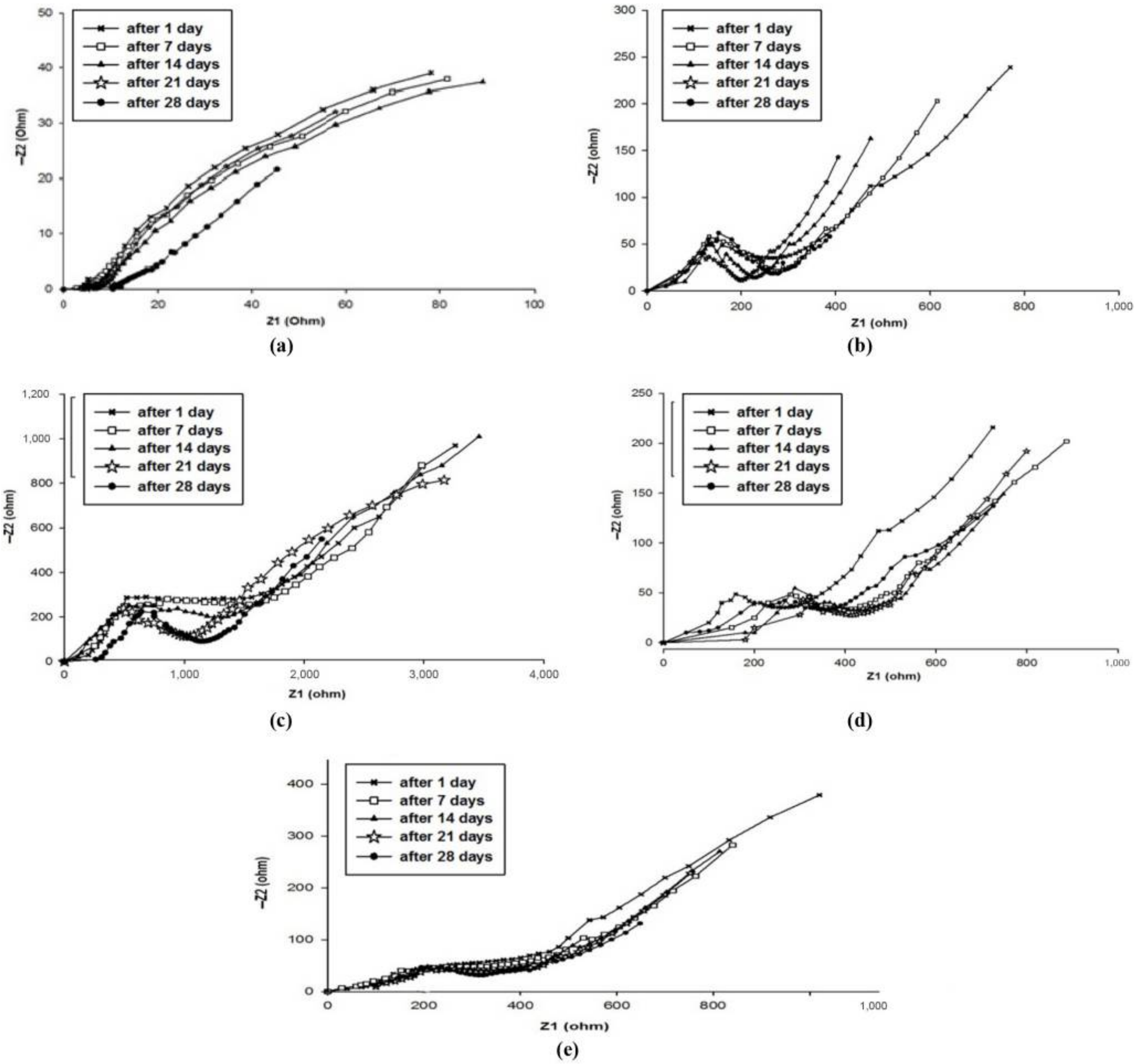
showed the highest Rp values among other specimens, which can be attributed to the presence of zinc phosphate in the shell that can perform various protection mechanisms through its sacrificial role and its high ionization rate to liberate zinc ions, which are well-known with their high ability in the inhibition of anodic reactions compared to other ions.

From the results of electrochemical measurements, it can be concluded that the presence of Si-Ph pigments in the coating enhances the corrosion protection of the rebars. The similar results obtained from both concrete and concrete admixtured with silica fume confirmed that the Si-Ph pigments can play both roles as anticorrosive pigments in coating formulations and as admixture in concrete, even if it is only present in the coating.

This can be attributed to the silica fume (core) that is found in the coating films with its high silicon dioxide content and very fine spherical particles, as well as high pozzolanic activity. Thus, even if it is found as a coating material, it can reserve its pozzolanic activity by chemically reacting with calcium hydroxide liberated from cement reaction, forming calcium silicate hydrate, which is considered as additional binder helping in densifying the inorganic concrete layer around the steel surface; this in turn increases its resistance to corrosion.

The slight higher corrosion resistance found in the concrete admixtured with silica fume can reveal this theory, besides that silica fume particles in the concrete matrix can decrease the porosity by filling its pores, allowing only small amount of corrosive materials to reach the steel surface (kelestemur and Demirel, 2010; Jolivet *et al.*, 2007; Hao *et al.*, 2013). Consequently, concrete containing coated rebars with the core-shell pigments having silica fume can lower the percentage of

Figure 7 EIS measurements of coated rebars with paints containing core-shell pigments vs time in 3.5 per cent NaCl



Notes: (a) Blank (uncoated); (b) coating containing silica fume (Si); (c) coating containing Si-ZnPh; (d) coating containing Si-SrPh; (e) coating containing Si-Zn.SrPh

silica fume admixtured with concrete (i.e. the amount can be less than 10 per cent in case of using Si-Ph pigments (Figure 9).

Case II

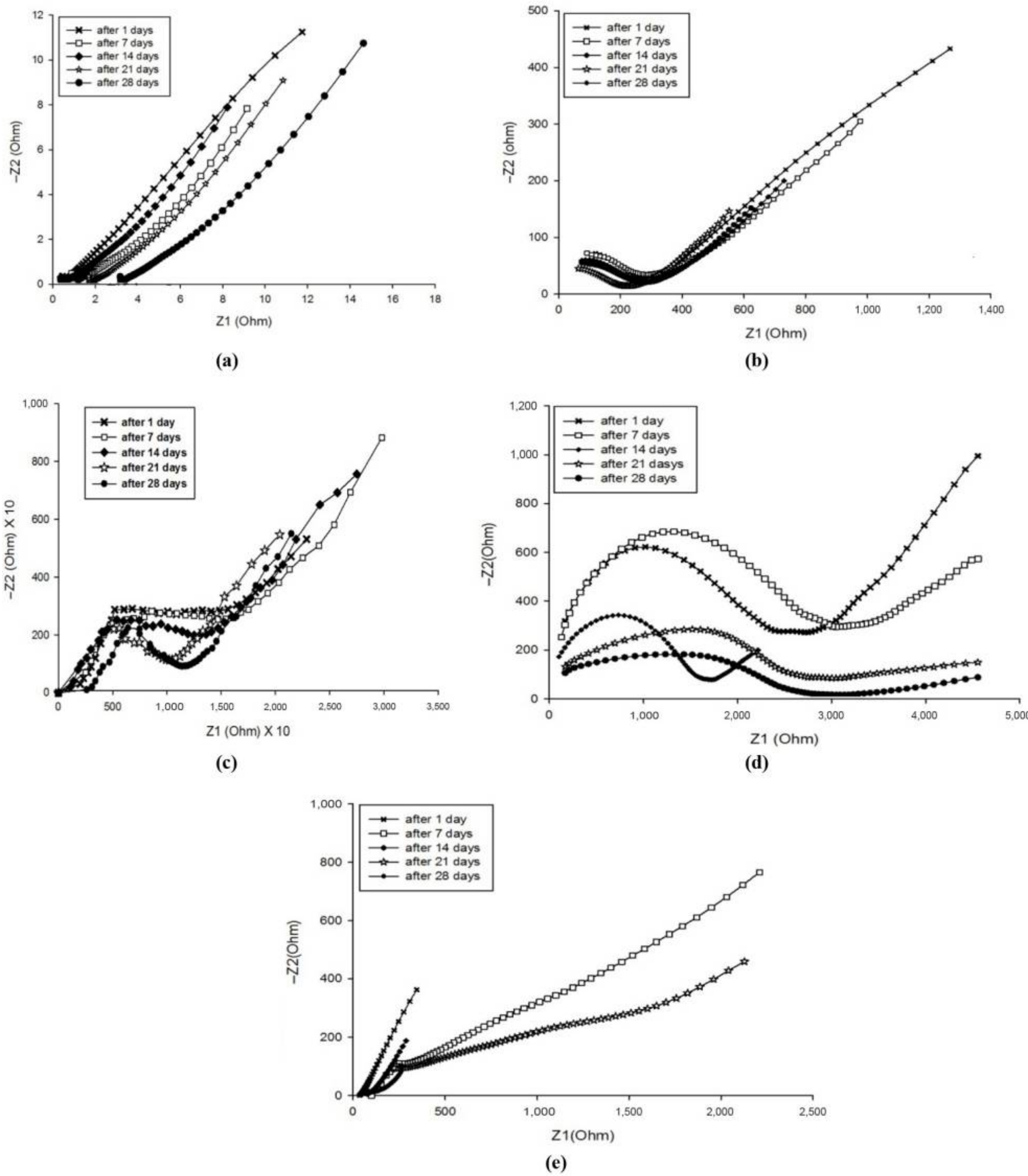
The second study in this work is to compare the corrosion efficiency of films containing Si-Zn, Sr and Zn.Sr phosphate (Si-Ph) pigments to those containing phosphate pigments in anticorrosive paint formulations.

Open-circuit potential

From Figure 10, it is obvious that all the formulations containing core-shell and phosphate pigments showed lower corrosion probability than uncoated rebar. This is may be due to the following issues:

- Coatings containing core-shell and phosphate pigments showed almost the same behavior during the immersion time by showing a dramatic drop in their potential due to the high chloride attack and breakdown of the passive film on the rebar surface in the first week. Then, the potential started to be stable due to the passivation process, depending mainly on the cations (Zn, Sr and Zn.Sr), which determine the mechanism of protection.
- Coatings having phosphates (Zn, Sr and Zn.Sr) showed slightly higher efficiency.

Figure 8 EIS measurements of coated rebars with paint containing core-shell pigments vs time in 3.5 per cent NaCl (concrete admixture with silica fume)



Notes: (a) Blank (uncoated); (b) coating containing SiO_2 (Si); (c) coating containing Si-ZnPh; (d) coating containing Si-SrPh; (e) coating containing Si-Zn.SrPh

Table V EIS results of coated rebar with paints containing core-shell pigments vs time in 3.5% NaCl

Time	Rp (Ohm · cm^2)
Blank	
1 day	2.8 × 10
7 days	2.904 × 10
14 days	3.29 × 10
21 days	2.979 × 10
28 days	1.82 × 10
Coating containing silica fume(Si)	
1 day	2.059 × 10^2
7 days	1.344 × 10^2
14 days	1.044 × 10^2
21 days	8.46 × 10
28 days	1.324 × 10^2
Coating containing Si-ZnPh	
1 day	2.541 × 10^3
7 days	2.159 × 10^3
14 days	5.990 × 10^2
21 days	9.689 × 10^2
28 days	8.668 × 10^2
Coating containing Si-SrPh	
1 day	1.961 × 10^2
7 days	1.794 × 10^2
14 days	1.651 × 10^2
21 days	1.372 × 10^2
28 days	2.011 × 10^2
Coating containing Si-Zn.SrPh	
1 day	2.488 × 10^2
7 days	2.25 × 10^2
14 days	1.942 × 10^2
21 days	2.080 × 10^2
28 days	2.145 × 10^2

- Coatings containing core-shell pigments exhibited slight lower corrosion resistance than those containing phosphate pigments. Hence, they are still considered as efficient anticorrosive pigments, taking in consideration that the core-shell contains only 10-20 per cent phosphate in their composition (as thin shell).

The good corrosion resistance of silica fume-phosphate Si-Ph pigments can be declared through different hypotheses.

The arrangement of these pigments in the paint film offer high corrosion protection by the reaction of silica fume with the cement component around the coated steel (pozzolanic activity).

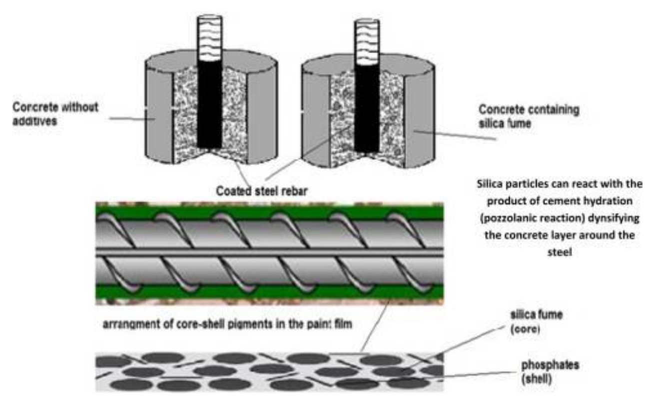
Phosphate shells are in direct contact with the surrounding media, and it is well known that these pigments can protect steel via different protection mechanisms such as the barrier effect offered by the plate structure of these particles and their ionization that leads to the blocking of the anodic areas found on the steel surface (Heydarpour *et al.*, 2014; Caprarim *et al.*, 2000).

The plate structures of phosphate pigments found in between spherical plates can block the interstices spaces between these spherical particles, leaving no route for the corrosive materials to reach the rebar surface.

Table VI EIS results of coated rebar with paints containing core-shell pigments vs time in 3.5% NaCl (concrete admixture with silica fume)

Time	Rp (Ohm · cm^2)
Blank	
1 day	3.82 × 10
7 days	4.24 × 10
14 days	4.16 × 10
21 days	3.71 × 10
28 days	3.25 × 10
Coating containing silica fume (Si)	
1 day	2.32 × 10^2
7 days	3.02 × 10^2
14 days	2.99 × 10^2
21 days	3.31 × 10^2
28 days	3.54 × 10^2
Coating containing Si-ZnPh	
1 day	*177.02*
7 days	*144.0*
14 days	*96.02*
21 days	*388.13*
28 days	*355.02*
Coating containing Si-SrPh	
1 day	3.14 × 10^3
7 days	2.943 × 10^3
14 days	1.88 × 10^3
21 days	1.76 × 10^3
28 days	2.21 × 10^3
Coating containing Si-Zn.SrPh	
1 day	3.60 × 10^2
7 days	8.20 × 10^2
14 days	3.53 × 10^3
21 days	9.15 × 10^2
28 days	8.91 × 10^2

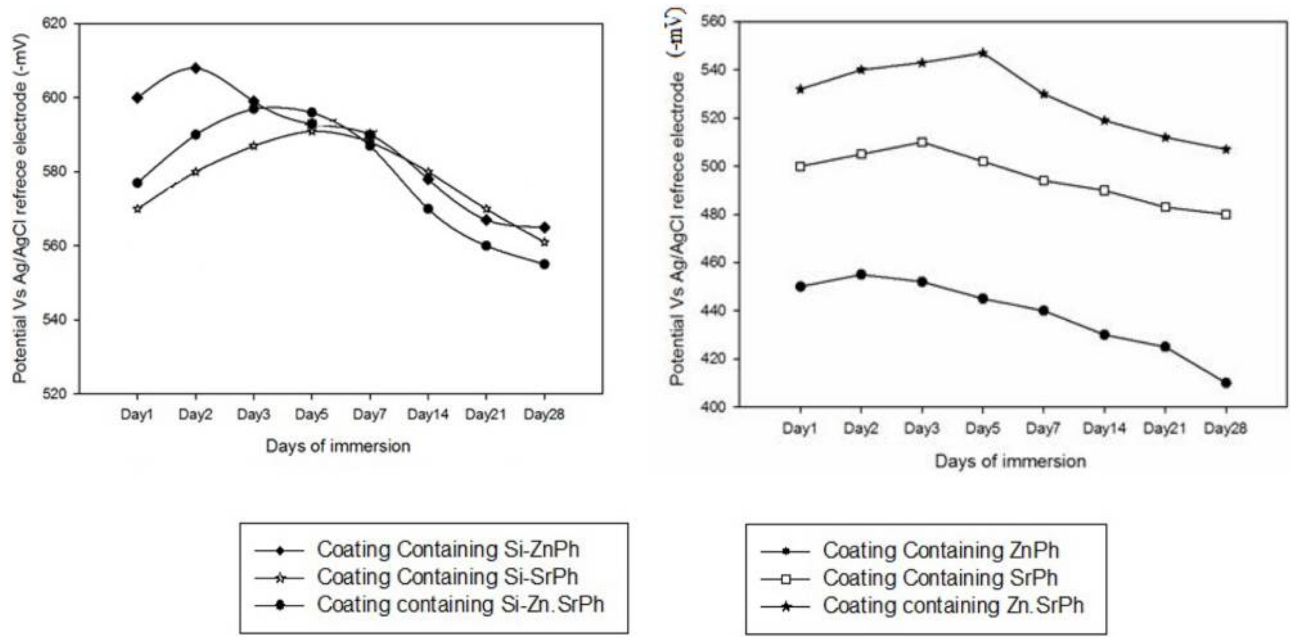
Figure 9 Schematic showing the arrangement of core-shell pigments in the paint film



Bond strength (pull out test)

After the evaluation of anticorrosive behavior of coatings containing core-shell pigments and comparing their behavior to phosphates, bond strength is another important test that is done to

Figure 10 Open-circuit potential for coatings containing phosphates and core-shell pigments in 3.5 per cent NaCl



estimate the effect of the silica fume-phosphate pigments on the bond strength between the coated rebars and the concrete cover.

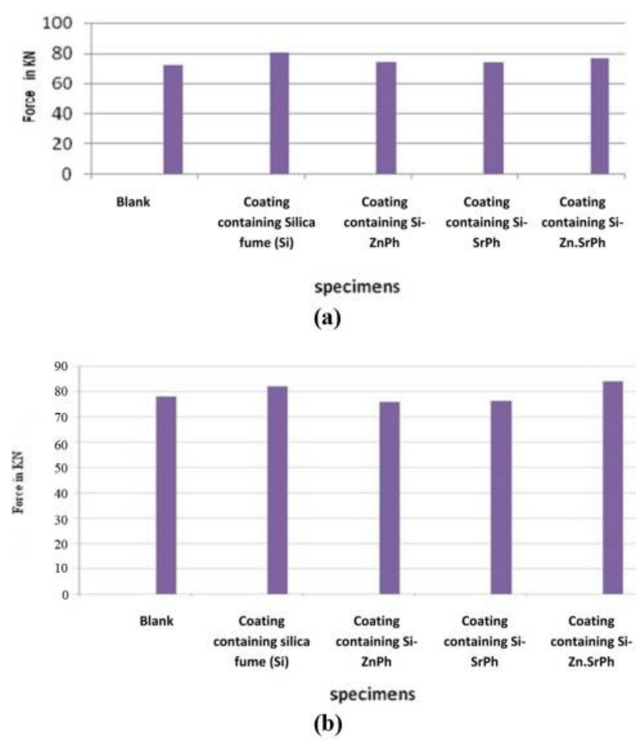
It is widely known that epoxy-based coatings used for concrete rebars may lower the bond strength between concrete and rebar because of the smooth and flat surfaces of these coatings. Also, considering the fact that epoxy coatings are organic in nature and the constituent elements of concrete are inorganic, it can be supposed that the interaction between the two materials of different nature may not be as strong as if they were of the same type (e.g. inorganic).

Pull out tests, shown in Figure 11, proved that different coated and uncoated samples in concrete and concrete admixtured with silica fume have negligible differences, which means that the used coating has not affected the bond between the rebar and concrete, and at the same time, the coating materials contribute to the perfection of the bond between them. This reveals that the coatings containing core-shell pigments can perform the same effect as that of the silica fume found in concrete as admixture, and they can show their pozzolanic activity even if it is found only in the coating. This is also clear in the results of bond strength of coating containing silica fume which offered higher bond strength than other coated samples. The obtained results in both cases confirmed the good performance of these coatings (Mohammadi *et al.*, 2014; Dotto *et al.*, 2004).

Conclusions

In this study, the effect of coatings containing new silica fume-phosphates core-shell pigments on the corrosion resistance and bond strength of rebars was investigated. The coated bars with core-shell pigments reinforced in concrete and concrete admixtured with silica fume were compared to study the effect of silica fume found in coating as pigment and in concrete as an admixture.

Figure 11 Bond strength (pull out test)



Notes: (a) Rebars in concrete; (b) rebars in concrete admixture with 10 per cent silica fume

The following results are obtained:

- The presence of core-shell pigments (based on 80-90 per cent of its structure on silica fume) in coatings can play the same role as that observed by adding silica fume in concrete as an admixture.

- The coating containing core-shell pigments did not affect the bond strength between rebar and concrete cover as it is convention in epoxy coatings, but in some cases, it offered higher bond strength.
- The comparison between the corrosion resistance and mechanical properties of coatings containing core-shell pigments and coatings containing phosphates estimated that coatings containing core-shell pigments can offer almost the same performance as those of phosphates.

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