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Satbayev University

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EVALUATION OF PROTECTIVE PROPERTIES OF COATINGS OF FILLED COMPOSITIONS BY ELECTROCHEMICAL METHODS

Abstract. This article discusses the possibility of using new chemically resistant fillers from local, non-scarce and inexpensive raw materials. It is known that mineral fillers are characterized by high chemical resistance. At the same time, glass powder has high acid resistance, and expanded clay dust has high acid and alkali resistance. The introduction of fillers into a heat-resistant anticorrosive coating based on ED-16 epoxy resin to protect metal products and structures from corrosion, contributes to improving the technological and operational properties of coatings. It is shown that on a liquid glass binder, with an increase in the dosage of the filler – glass powder, the adhesive strength of coatings and its protective ability increases. When used as a filler of expanded clay dust, on the contrary, with an increase in the dosage of the filler, the adhesive strength of the coatings and its protective ability decrease. The introduction of expanded clay dust improves the electrochemical state of the steel under the coating, and at the same time increases the fire resistance of the coating, which significantly reduces the deformation of the metal when exposed to high temperature (up to 900-950°C). Having a large surface, the fillers come into contact with the functional groups of the polymer, while the adhesion forces are manifested, the nature of which determines the resistance to aging of coatings and the preservation of their protective properties for a long time. The influence of glass powder and expanded clay dust on the adhesive properties and protective ability of coatings has been studied. It is known that visual assessment of the corrosion state of steel samples does not provide accurate information about the further electrochemical state of steel samples under coating. Therefore, electrochemical studies of steel samples protected by anticorrosive coatings have been carried out by removing anodic polarization curves, as well as by measuring the ohmic resistance of coatings. It is concluded that the data obtained by capacitance-ohmic measurement are similar to the data obtained from the anode polarization curves of steel samples protected by anticorrosive coatings. Tests have shown that the greatest displacement of the stationary potential is observed when filling the binder with glass powder, regardless of the type of binder.

Key words: anticorrosive coatings, filler, glass powder, steel, metal structures.

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ЖАБЫЛҒАН ҚҰРАМДАРДЫҢ ҚОРҒАНЫШТЫҚ ҚАСИЕТТЕРІН ЭЛЕКТРОХИМИЯЛЫҚ ӘДІСТЕРМЕН БАҒАЛАУ

Аннотация. Бұл мақалада жергілікті, тапшы емес және қымбат емес шикізаттан жаңа химиялық төзімді толтырғыштарды пайдалану мүмкіндігі қарастырылады. Минералды толтырғыштардың химиялық төзімділігі жоғары екені белгілі. Сонымен бірге шыны ұнтағының қышқылға төзімділігі жоғары, ал керамзит шаңының қышқыл мен сілтіге төзімділігі жоғары. Металл бұйымдары мен конструкцияларын коррозиядан қорғау үшін ED-16 эпоксидті шайыр негізіндегі ыстыққа төзімді коррозияға қарсы жабынға толтырғыштарды енгізу жабындардың технологиялық және пайдалану қасиеттерін арттыруға ықпал етеді. Сұйық шыны байланыстырғышта толтырғыш – шыны ұнтағының мөлшерлемесі ұлғайған сайын жабындардың адгезиялық беріктігі мен оның қорғаныс қабілеті арта түсетіні көрсетілген. Толтырғыш ретінде пайдаланылғанда, керамзит шаңы, керісінше, толтырғыштың дозасының жоғарылауымен, жабындардың адгезиясының беріктігі және оның қорғаныс қабілеті төмендейді. Керамзит шаңын енгізу жабын астындағы болаттың электрохимиялық күйін жақсартады және сонымен бірге жабынның отқа төзімділігін арттырады, бұл жоғары температура әсер еткенде металдың деформациясын айтарлықтай төмендетеді (дейінгі диапазонда). 900-950°C).

Үлкен бетке ие бола отырып, толтырғыштар полимердің функционалды топтарымен байланысқа түседі және адгезия күштері көрінеді, олардың табиғаты жабындардың қартаюына төзімділігін және ұзақ уақыт бойы олардың қорғаныс қасиеттерін сақтауды анықтайды. Шыны ұнтағы мен керамзит шаңының жабындардың адгезиялық қасиеттері мен қорғаныс қабілетіне әсері зерттелді. Болат үлгілерінің коррозиялық күйін визуалды бағалау жабын астындағы болат үлгілерінің одан әрі электрохимиялық күйі туралы нақты ақпарат бермейтіні белгілі. Сондықтан коррозияға қарсы жабындармен қорғалған болат үлгілеріне электрохимиялық зерттеулер анодтық поляризация қисықтарын өлшеу, сонымен қатар жабындардың омдық кедергісін өлшеу арқылы жүргізілді. Сыйымдылық-омдық өлшеуде алынған нәтижелер коррозияға қарсы жабындармен қорғалған болат үлгілерінің анодтық поляризация қисық сызығын алу арқылы алынған мәліметтерге ұқсас деген қорытынды жасалды. Сынақтар стационарлық потенциалдың ең үлкен ығысуы байланыстырғыштың түріне қарамастан шыны ұнтағымен толтырылған кезде байқалатынын көрсетті.

Түйін сөздер: коррозияға қарсы жабындар, толтырғыш, шыны ұнтақ, болат, металл конструкциялар.

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ОЦЕНКА ЗАЩИТНЫХ СВОЙСТВ ПОКРЫТИЙ НАПОЛНЕННЫХ КОМПОЗИЦИЙ ЭЛЕКТРОХИМИЧЕСКИМИ МЕТОДАМИ

Аннотация. В данной статье рассматривается возможность применения новых химически стойких наполнителей из местных, недефицитных и недорогих сырьевых материалов. Известно, что минеральные наполнители отличаются высокой химической стойкостью. При этом стеклянный порошок имеет высокую кислотостойкость, а керамзитовая пыль обладает высокой кислото- и щелочестойкостью. Введение наполнителей в термостойкое антикоррозионное покрытие на основе эпоксидной смолы ЭД-16 для защиты металлических изделий и конструкций от коррозии, способствует повышению технологических и эксплуатационных свойств покрытий. Показано, что на жидкостекольном связующем с повышением дозировки наполнителя – стеклянного порошка повышается адгезионная прочность покрытий и его защитная способность. При применении в качестве наполнителя керамзитовой пыли, наоборот, с повышением дозировки наполнителя снижается адгезионная прочность покрытий и его защитная способность. Введение керамзитовой пыли улучшает электрохимическое состояние стали под покрытием и одновременно повышает огнестойкость покрытия, что значительно снижает деформацию металла при воздействии на нее высокой температуры (в пределах до 900-950°С). Обладая большой поверхностью, наполнители вступают в контакт с функциональными группами полимера, при этом проявляются силы адгезии, природа которых обусловливает стойкость к старению покрытий и сохранение в течение длительного времени их защитных свойств. Изучено влияние стеклянного порошка и керамзитовой пыли на адгезионные свойства и защитную способность покрытий. Известно, что визуальная оценка коррозионного состояния стальных образцов не дает точной информации о дальнейшем электрохимическом состоянии стальных образцов под покрытием. Поэтому проведены электрохимические исследования защищенных антикоррозионными покрытиями стальных образцов методом снятия анодных поляризационных кривых, а также измерением омического сопротивления покрытий. Сделан вывод о том, что полученные при емкостно-омическом измерении сходны с данными, полученными снятием анодных поляризационных кривых стальных образцов, защищенных антикоррозионными покрытиями. Испытания показали, что наибольшее смещение стационарного потенциала отмечается при наполнении связующего стеклянным порошком независимо от вида связующего.

Ключевые слова: антикоррозионные покрытия, наполнитель, стеклянный порошок, сталь, металлические конструкции.

Introduction. Steel and its alloys are used in all leading industries and construction, but these materials are highly sensitive to the effects of various types of corrosion. Currently, Russia produces anticorrosive coating compositions based on imported raw materials. The largest manufacturers of epoxy polymers for anticorrosive coatings were Dow Chemical, Shell, Nan Ya. EHB insulation systems provide corrosion protection in a wide variety of conditions, as thanks to a special technology, anticorrosive coatings such as Ceramite Glassguard have high performance, durability and versatility. Coatings of this type contain millions of flakes of heat-cured glass in various resins. These flakes self-cleave in the coating shortly after application and are completely encapsulated, forming a strong barrier capable of withstanding the effects of any corrosive environment. (Leviev et al., 2021:5).

The components consist of a mixture of oxidized petroleum jelly, microcrystalline waxes, metal sulfonates, water-displacing agents, and pigments in a hydrocarbon solvent. The components of oxidized petroleum jelly and microcrystalline waxes have the ability to remove moisture from the surface, and sulfonates form an absorbent layer of corrosion inhibitor on metal surfaces (Mammedov et al., 2021:8).

Analysis of literature sources shows that for corrosion protection of metal structures and equipment, an important role is assigned to fillers that are introduced into the binder to reduce shrinkage of coatings, increase their strength and protective ability in various aggressive environments, as well as to reduce the cost of coatings on both liquid-glass binder and polymer.

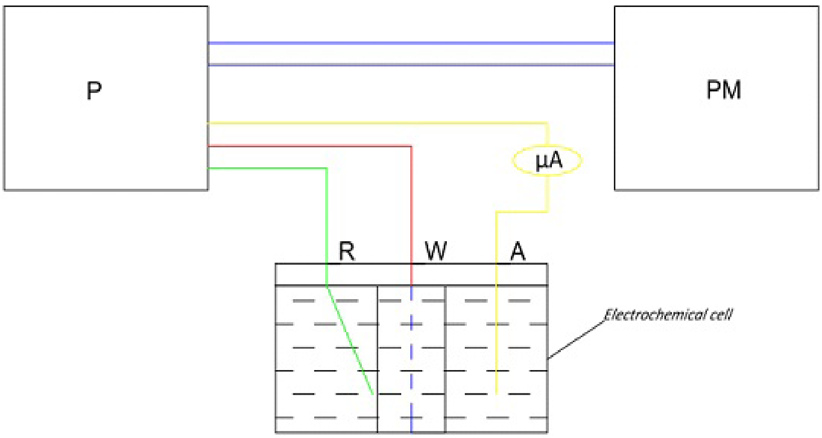
Materials. For the preparation of anticorrosive composite coatings for the protection of metal structures, epoxy compounds may be suitable as a binder along with liquid glass. However, they are prone to shrinkage, and are not elastic enough, which causes fears of cracks during transportation and installation of metal structures. Therefore, it becomes necessary to modify the polymer with special additives. It is known from the literature that various plasticizers are used to give elasticity to coatings based on epoxy resins. In our experiments, we adopted dibutyl phthalate as a plasticizer, the most available and effective plasticizer.

It is known that metal structures, when exposed to high temperatures commensurate with the temperature during a fire, can deform, and the epoxy coating can burn out, and thereby lose protective functions. In order to increase the heat resistance of the anticorrosive coating, we modified it with the addition of a mineral filler - expanded clay dust.

From the practice of selecting paintwork compositions, it is known that their protective properties improve with the introduction of fillers (Kovrizhkina et al., 2019:8) which, in addition, improve the film formation process, reduce residual stresses, and change the electrochemical state of steel under coating (Shintemirov et al., 2009:4). Non-deficient fillers, ground glass fight and expanded clay dust were used for experiments.

Methods. When metal comes into contact with an anticorrosive coating, metal ions are transferred into the coating, as well as when unprotected metal comes into contact. In this case, metal ions pass into the coating, similar to how it happens in true electrolyte solutions. An unbalanced number of electrons remains in the metal, therefore, a double electric layer is formed at the metal-coating boundary (Rossina et al., 2019), which has its own potential. It also happens under a polymer coating. In that way, if you connect a coated metal and a coating into a circuit, then a current will flow through it, which is called a corrosion current. Consequently, a corrosion process occurs on the metal under the coating. The course of this process was controlled by potentiostatic and capacitance-ohmic (Rossina et al., 2019) measurements of the corrosion current in samples subjected to corrosion tests.

To remove the anode polarization curves, we used the IPC-PRO potentiostat. Metal samples with coatings (working electrodes), a silver chloride reference electrode, an auxiliary platinum electrode (Yaroslavtseva et al., 2015) were immersed in a dielectric vessel with an aggressive solution (electrochemical cell) and connected to a potentiostat according to the scheme (Figure 1).

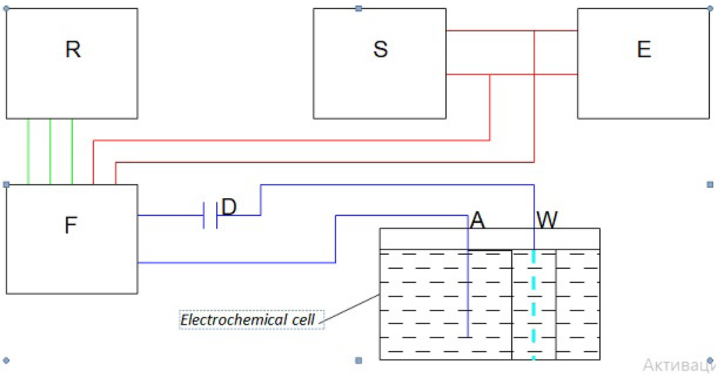


P - potentiostat; PM - potentiometer; µA - microammeter; R - reference electrode; W - working electrode; A - auxiliary electrode

Figure 1. − Connection diagram of devices for conducting potentiostatic studies

The recording of potentials and polarization currents was carried out with a HANS ORION potentiometer, as well as with an M254 microammeter. When selecting the composition, the potentiostatic method made it possible to select the most effective passivating ingredients. Their effectiveness is most evident with small coating thicknesses (up to 50 microns) (Mammedov et al., 2021:8).

When studying coatings with a thickness of more than 50 microns, when they have high dielectric and barrier properties, the capacitive-ohmic method was used. To measure capacitance and ohmic resistance, coated samples were placed in an electrochemical cell. To make electrical contact with an electrically conductive body of a water-saturated anticorrosive coating, an auxiliary electrode made of platinum was placed in an electrochemical cell (Yaroslavtseva et al., 2015) and through a separating capacitor of sufficiently large capacity, which did not affect the measurement results, the sample was connected to a full resistance meter (Figure 2). A recorder was used to record the measured parameters, and the frequency ($\omega = 10^4$) was set by an audio frequency generator and controlled by a frequency meter.



F - full resistance meter; R - recorder; S - sound frequency generator; E - electronic counting frequency meter; W - working electrode; A - auxiliary electrode; D - decoupling capacitor.

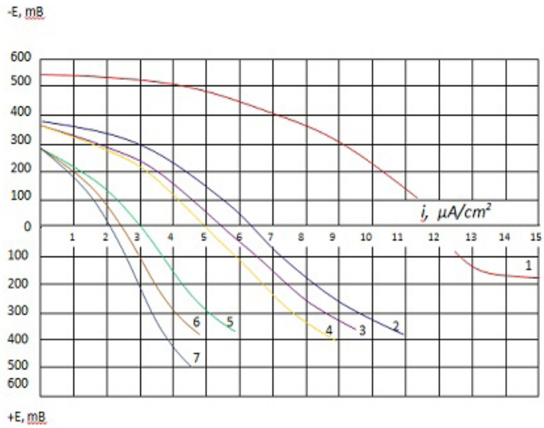
Figure 2. - Connection diagram of devices for conducting capacitance-ohmic studies

Results. It is known that visual assessment of the corrosion state of steel samples does not provide accurate information about the further electrochemical state of steel samples under coating. In addition, when visually assessing the corrosion condition, it is necessary to remove the coating from the surface of steel samples, which is fraught with considerable difficulties, since after removing the coating itself, the samples still have to be etched with hydrochloric acid inhibited by urotropin. This process can additionally cause corrosion of steel, under the influence of hydrochloric acid. Therefore, electrochemical studies of steel samples protected by anticorrosive coatings have been carried out by removing anodic polarization curves, as well as by measuring the ohmic resistance of coatings.

Figures 3 and 4 show the results of electrochemical studies of the condition of steel samples protected by anticorrosive coatings based on liquid glass and epoxy resin. The studies were carried out by the potentiostatic method, by removing the anode polarization curves on the IPC-PRO potentiostat. The dependence of the polarization current density on the potential was recorded with a ZGANS ORION potentiometer. The auxiliary electrode was a 12X18H10T stainless steel ring. The reference electrode was a silver chloride electrode filled with a saturated solution of KCl.

In the case of using glass powder as a filler, the corrosion current density decreases even more and ranges from 3.2 to 5.0 μA/cm^2, depending on the thickness of the coating, which in turn depends on the amount of filler introduced (Figure 3). These values of the polarization current density reduction indicators indicate full protection of the steel sample with tested coatings.

Figure 3 also shows that anticorrosive coatings shift the stationary potentials of the steel sample in the positive direction by 120-150 mV when filling the liquid-glass binder with expanded clay dust. If this binder is filled with glass powder, then the displacement of the stationary potential is 220-250 mV.



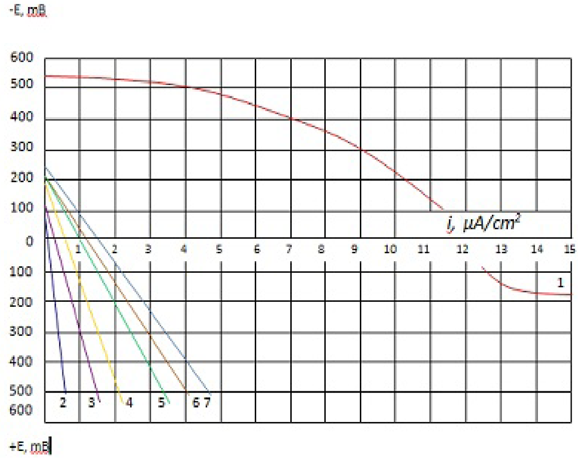
1 – uncoated sample after 180 cycles; 2 - coated sample, where the filler was expanded clay dust (100% of the mass of liquid glass); 3 – the same (125% of the mass of liquid glass); 4 - the same (150% of the mass of liquid glass); 5 – the same filler glass powder (100% by weight of liquid glass); 6 - the same filler glass powder (125% by weight of liquid glass); 7 - the same filler glass powder (150% by weight of liquid glass).

Figure 3 - Anodic polarization characteristics of steel with anticorrosive coatings on a liquid-glass binder after 180 cycles of accelerated corrosion tests

When using an anticorrosive coating based on a liquid-glass binder, the average specific polarizability of samples increases (from 40.1-47.7 to 69.7-89.0 mV /μA /cm^2), and the stationary potential of steel is shifted by the coating in the positive direction (by 110 - 160 mV) when used as fillers of expanded clay dust.

Somewhat greater polarizability of the steel sample is observed when using glass powder as fillers. At the same time, the average specific polarizability increases from 66.6-92.0 to 115.4-140.0 mV/μA/cm^2.

Figure 4 shows the anodic polarization curves of a steel sample protected by an anti-corrosion coating based on epoxy resin, where expanded clay dust and glass powder are used as fillers.



1 – uncoated sample after 180 cycles; 2 - coated sample, where the filler was expanded clay dust (100% by weight of epoxy resin); 3 – the same (125% by weight of epoxy resin); 4 - the same (150% by weight of epoxy resin); 5 – the same filler glass powder (100% by weight of epoxy resin); 6 - the same filler glass powder (125% by weight of epoxy resin); 7 - the same filler glass powder (150% by weight of epoxy resin).

Figure 4 - Anodic polarization characteristics of steel with anticorrosive coatings based on epoxy resin after 180 cycles of accelerated corrosion tests

Figure 4 shows that anticorrosive coatings based on epoxy resin reliably protect steel samples from corrosion, regardless of which filler was used in the coating. However, the analysis of the curves shows that in the case of filling the epoxy resin with glass powder, the values of the polarization current densities are less than when filling the resin with expanded clay dust.

Table 1 shows the results of tests of anticorrosive coatings by the capacitive-ohmic method. These data are necessary for the correct interpretation of the obtained measurement results of capacitance (C) and ohmic resistance (R), which depend not only on the properties of the coatings at the time of measurements, but also on the frequency of the current at which these measurements are made. In addition, measurements of capacitance and ohmic resistance objectively complement the results obtained by removing the anode polarization curves of steel samples protected by anticorrosive coatings (Yaroslavtseva et al., 2016).

Table 1 - Effect of filler additives on the electrochemical properties of protective coatings

Type of filler	Stationary electrode potential, mV	Shift of stationary potential of coated steel, mV	Polarization current density, μA/cm^2	Average specific polarizability of the coated reinforcement, mV/μA/cm^2	Number of test cycles
Anticorrosive coating based on liquid glass					
glass powder	-250…280	270…300	3,2…5,0	56…93,75	180
expanded clay dust	-136…170	170…210	7,5…9,4	22,6…22,3	180
Anti-corrosion coating based on epoxy resin					
glass powder	-75…100	450…475	0,5…1,4	71,4…150	180
expanded clay dust	-170…210	340…380	2,4…3,4	61,8…70,8	180
control (uncoated)	-550	-	25,7	-	180

It can be seen from Table 1 that the data obtained by capacitance-ohmic measurement are similar to the data obtained from the anode polarization curves of steel samples protected by anticorrosive coatings.

Discussion. Epoxy resin-based coatings filled with expanded clay dust reduce the polarization current density at a potential of +300 mV (from 25 to 2.2-3.2 μA /cm^2), which is more than three times more effective than anticorrosive coatings based on liquid glass. In the case of using glass powder as a filler, the corrosion current density decreases even more and ranges from 0.5 to 1.5 μA/cm^2, depending on the thickness of the coating, i.e. the protective ability increases with increasing coating thickness. These values of current density reduction indicators indicate full protection of the steel sample with tested coatings. The average specific polarizability of the samples increases from 68.75 to 120.0 for coatings where the filler was expanded clay dust and up to 90-100, where the filler was glass powder. The presented experimental results show that the specific polarizability of steel samples protected by anticorrosive coatings based on epoxy resin is almost 1.5 - 2 times higher than samples protected by coatings based on liquid glass. Tests have shown that the greatest displacement of the stationary potential is observed when filling the binder with glass powder, regardless of the type of binder. However, even in this case, it is impossible to discount such a filler as expanded clay dust, which also reliably protects steel samples from corrosion. In that way, based on preliminary experiments, fillers – glass powder and expanded clay dust from the cold end of the firing furnace - were selected as components of an anticorrosive coating based on liquid glass and epoxy resin.

Conclusion. Analysis of the anodic polarization characteristics taken after

180 cycles of accelerated corrosion tests shows that the protection of steel samples with anticorrosive coatings based on liquid glass filled with expanded clay dust or glass powder, in all cases:

- improves the electrochemical condition of steel;
- the polarization current density decreases at a potential of +300 mV (from 25 to 7.0-9.2 μA/cm^2) when using expanded clay dust as a filler.

However, the analysis of the curves shows that in the case of filling the epoxy resin with glass powder, the values of the polarization current densities are less than when filling the resin with expanded clay dust.

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