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Comprehension of demoulding mechanisms at the formwork/oil/concrete interface

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Abstract The implementation of concrete and its association with a release agent influence the aesthetics of the concrete facings. The mineral oils tend to be replaced by vegetable formulations, to reduce the impact of the substances spilled in the environment. From a technical point of view, it is important to characterize the action of these new formulations at the interface concrete/oil/formwork. Two performing techniques have been used to study the physico-chemical processes, the tribometry and electrochemical impedance spectroscopy. The correlation of the results obtained allowed to improve the understanding of the mechanisms involved at the interface mould/oil, in connection with the use of an acidifier in the formulation.

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Résumé *La mise en œuvre du béton et son association avec un agent de démoulage, influence l'esthétique des parements en béton. Les huiles minérales tendent à être remplacées par des formulations à base végétale, afin de réduire l'impact des substances rejetées dans l'environnement. Sur un plan technique, il est important de caractériser le mode d'action de ces nouvelles formulations, à l'interface béton/huile/coffrage. Les travaux s'appuient notamment sur l'utilisation de deux techniques performantes pour étudier les phénomènes physico-chimiques, la tribométrie et la spectroscopie d'impédance électrochimique. La corrélation entre les résultats obtenus permet d'améliorer la compréhension des mécanismes impliqués à l'interface de démoulage, en relation avec l'utilisation d'un acidifiant dans la formulation.*

Keywords Concrete · Release agents · Acidifier · Interfacial mechanisms · Formwork

Mots clés béton · agents de démoulage · acidifiant · mécanisme interfacial · coffrage

1 Introduction

Evolving techniques for using concrete offer new possibilities to explore the aesthetics of facings [1].

A concrete facing is a formworked surface that is often visible after completion of the structure, and acts as a skin without defect (tint variations, bubbling or dust formation). It must withstand the various attacks sustained by the structure during its service life. Achieving a high level quality facing involves the control of several parameters :

– the type of concrete formulation,
– the conditions of implementation : quality of formwork walls, method of pouring and concrete vibrations,
– the type of release agents necessary to prevent the concrete from catching on the metal surface.

Mouldable concretes, in addition to their statutory physical qualities and mechanical performances, must also meet environmental requirements. The selection of bioproducts thus determines the compositions of both cement and release agents.

Among release agents available, we can find petrochemical-based products usually called mineral oils and vegetable-based formulations developed to improve the compatibility with environment [2]. Theses products can be prepared according to several types of formulations : neat oil, emulsion or blend with (bio)solvent.

If the performances of release oils were long neglected, their contribution to the quality of the concrete facing is now studied with care.

The present study aims to assess the performance of four "model" neat oils in order to identify the key parameters which are involved in the efficiency of the formulations.

Although the differences of chemical structure between the mineral oil and the vegetable oil, the addition of fatty acids is usually performed in both cases. It seems necessary to compare the effect of fatty acids at the interface oil/mould in order to optimize the compositions of the both formulations.

The interfacial properties of formulations depend on the following parameters:

- The mechanical friction generated by the concrete,
- The thickness and physical-chemical properties of the film,
- The interactions concrete/oil and oil/formwork wall.

Tribological behaviours of the formulations were compared by using a plane tribometer specially devised for studying such frictions due to the placement of cement in formwork. The coating properties of the films were assessed by electrochemical impedance spectroscopy.

2 Properties of release agents

For the present study, CHRYSO provided four neat oils, inspired of the marketed formulations, but containing only the two active compounds really influencing the demoulding efficiency (the base oil and the acidifier).

M0 refers to the mineral based oil. V0 represents the vegetable based oil corresponding to a blend of esters prepared from vegetable oils. They are ready-to-use delayed-action release agents. The mineral oil is a pale yellow liquid, the vegetable oil is a straw-yellow liquid.

M1 represents the primary base M0 with the acidifier.

V1 consists of the vegetable base V0 with the same percent of the acidifier.

The acidifier is mainly composed of oleic acid.

The oleochemical esters of V0 have the advantage to be safe products for the environment and the users' health.

The compositions of the formulations are given in Table 1.

The acid value is representative of the quantity of free fatty acid in the formulations. It corresponds to the number of milligrams of potassium hydroxide (KOH) required to neutralize one gram of oil.

Two reactions involving fatty acids are to be taken into account ;

Table 1 Characteristics of the formulations

	Vegetable based oils		Mineral based oils	
Reference	V0	V1	M0	M1
Acidity induce	IA <1 mg KOH/g prdt	IA = 6 mg KOH/g prdt	IA <1 mg KOH/g prdt	IA = 6 mg KOH/g prdt
Density	0.93	0.93	0.85	0.85
Viscosity (mPa.s)	177.7	178.3	20.4	27.6

- the fatty acids in presence of calcium hydroxide lead to a calcium carboxylate,
- the saponification between esters and calcium hydroxide also generates calcium carboxylate (Fig. 1). Saponification only occurs in the case of the vegetable formulation.

The polar head which has an affinity with water is hydrophilic. The chain has an affinity with a lipophile medium. In the presence of water, the carboxylate ions organize into aggregates or spherical micelle (Fig. 2).

3 Study of the oil behaviour at the interface

The conditions of application are more demanding for vegetable formulations than for mineral oils. Especially, vegetable formulations must be applied in a thin and continuous film, for an overdosing causes poor facing quality. Therefore, the development of application skills is a key to success

When the concrete is poured, the release agent is subjected to stress due to the friction of the concrete on the formwork surface. It is essential that the release agent withstands this disruption in order to prevent displacement of the oil film to the bottom of the formwork.

3.1 Concrete and metal-plate characteristics

The concrete used for the tests is a traditional B25 type. Its composition is given in Table 2. A slump test using the Abrams cone was conducted after each batch to check its workability. This is between 10 and 12 cm. The water/cement ratio was 0.51.

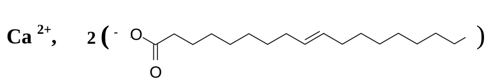


Fig. 1 Representation of calcium oleate

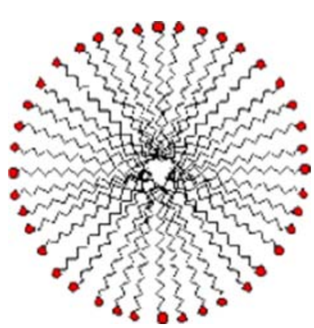


Fig. 2 Diagram of a micelle

Table 2 Concrete composition

Composition	kg/m3
Fine gravel 5/20	761
Fine gravel 4/10	281
Sand 0/4	826
Cement CEMI 52,5	258
Filer MEAC P2	86
Water	175
W/L	0.51

The concrete is made as follows: (Fig. 3)

The plate used for this study was cut out of a formwork wall by the manufacturer (steel E24 polished to remove calamine). Roughness was measured using a portable roughness meter:

Ra = 0.8 µm and Rt = 8 µm with Ra as the arithmetic mean of the profile deviations from the mean line, and Rt as the distance between the highest and the lowest points of the roughness profiles.

3.2 Tests of applications

The type of spraying equipment is essential to guarantee an equal wetting.

To simulate the application on site, the oils were applied on formwork with an ecospray sprayer equipped with a flat nozzle. The pressure was 4.5 bars and the distance with the surface 20 cm.

Two different qualities of film have been prepared;

- Application only by spraying the oil (spray time: 0.5 s),
- Application by spraying and then spreading the film with a rubber squeegee, to remove the surplus.

The thicknesses of the oil films were measured by PIXE analysis [3]. The values resulting from four measurements, are presented in table 3. For sprayed-on films, a value interval is more representative of the observed deviations.

The thickness of the oil film closely correlates with the viscosity of the oils mentioned in Table 1.

According to the micrographs of spray-on films, the mineral oils produce a consistent film whereas the

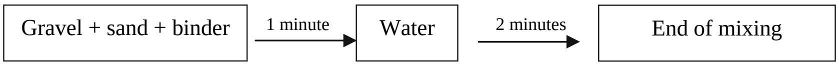


Fig. 3 Preparation of concrete

Table 3 Thicknesses of oil films measurec by particle induced X-ray emission (PIXE):precision:0.1 μm

Oil Nature	Thickness (μm) for pulverized film	Thickness (μm) for scraped film after pulverization
M0	[2–3]	1.2
M1	[3–5]	1
V0	[2.4–3.5]	2
V1	[2–3]	1.6

vegetable oils present juxtaposed droplets, mainly due to their higher viscosities. Several spays of vegetable formulations are necessary to cover the entire metal surface, but leads to an excess of oil on the formwork [4].

After scraping, the vegetable film is the most uniform and presents a thickness compatible with a high quality facing.

The influence of the two application methods are compared using a plane tribometer.

3.3 Tribometer tests

Figure 4 shows the experimental device for simulation of concrete-formwork friction during pouring. A plate travels between two concrete samples. It is moved by a motor coupled to a worm. The samples are pressurized using jacks. The concrete was placed in two 120 mm diameter sample holders. The seal is mounted on the sample-holders to prevent concrete

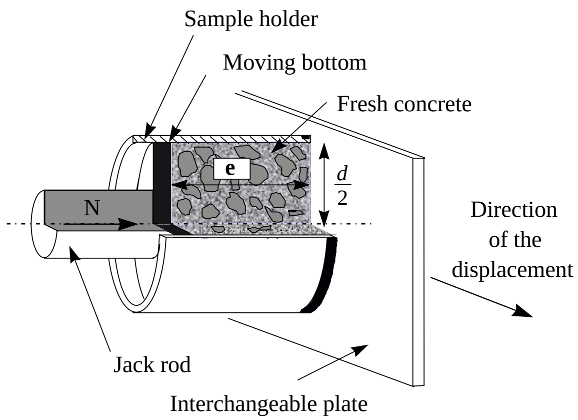


Fig. 4 Tribometer principle

setting without damaging the oil film applied to the plate [5].

The friction stress is calculated as follows:

$$\tau_f = \frac{\text{Measured force} - \text{Parasitic force}}{\text{Cross} - \text{sectional area of sample}} = \frac{F_{mes} - F_{par}}{Sc} \text{ (KPa)}$$

Sc is the surface area in contact with the sample on the plate (Sc = 113.1 cm^2) and F_{par} is the result of the parasitic friction forces caused by the seal against the plate. This value must be as low as possible.

The test pressure values correspond to the thrust range measured at the bottom of the formwork, i.e. 50, 100, 150 and 200 kPa. Plate displacement speed corresponds to the concrete pouring speed: 6 m/h, or 1.67 mm/s.

3.3.1 Results for a sprayed film

Despite its appearance, fluid concrete is not a continuous medium. Each ingredient has a specific role during friction.

The stress applied to the material is transmitted, during the granular phase and to the paste formed by the binder (cement, filler). This stress will cause part of the liquid phase and of the fines to migrate towards the interface. As a result, a lubricating limit layer comprising water and fines appears at the interface. Because the paste proportion is low (28%), the limit layer is not thick enough to prevent the gravel from coming in contact with the metal surface (Fig. 5). The friction will be granular.

Figure 6 shows the friction stress as a function of contact pressure. Friction stress increases linearly

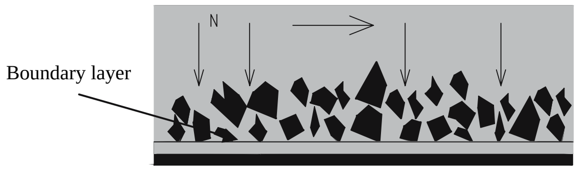


Fig. 5 Schematic representation of the phenomena related to the pressure

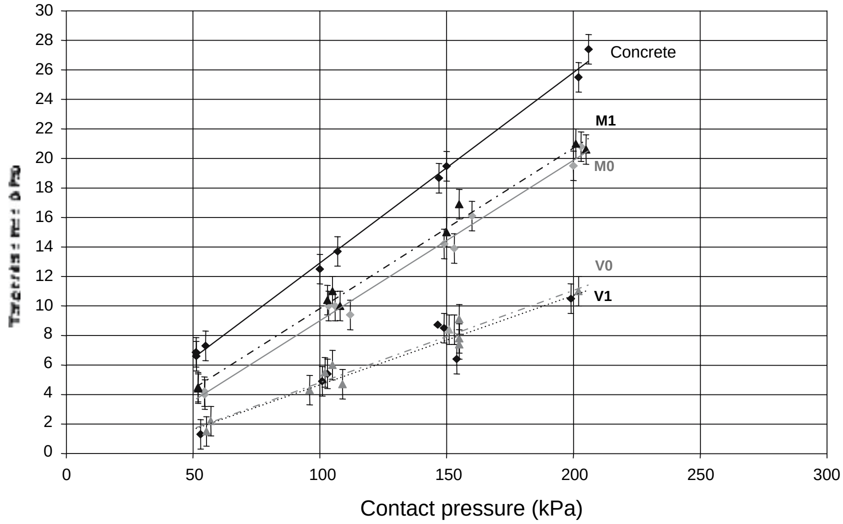


Fig. 6 Friction constraints versus contact pressure for pulverized mineral and vegetable oils

with contact pressure. In the pressure range studied, concrete friction follows Coulomb's law.

The friction stress values obtained for oils M0 and M1 are very similar. Given the uncertainty of the measurements, their behaviours in contact with the concrete are close. The fall in friction stress compared to the concrete is of about 10%.

For vegetable oils, friction stresses are lower than those obtained for the mineral oils. The curve slope as a function of pressure differs from those obtained for the concrete and for the mineral oils, meaning that the release mechanism at the concrete-formwork interface is different. Friction stress falls by about 45%.

The addition of acidifier in the formulation M0 and V0 has no clear influence on the friction stresses.

3.3.2 Results for a spread film

Figure 7 shows the measurements conducted on the oil films that were sprayed-on and then spread.

Adding an acidifier has still no effect on the tribological behaviour of the mineral oils. However, with the vegetable oils, the acidifier causes a substantial drop in friction stress compared to the concrete.

The reduction in friction stress rises to 10% in the case of mineral oils, and to 30% and 45% in the case of the vegetable oils V0 and V1.

3.3.3 Comparison of the two application methods

With an application by spray, the reduction in friction is governed by the thickness of the film.

The increase of thickness leads to a proportional reduction in friction stress compared to concrete-wall friction stresses. The action of acidifier is not significant on the reduction of friction.

For a spread film, the role of acidifier becomes predominant. This chemical action then leads to a decrease of friction from V0 to V1.

4 Electrochemical impedance spectroscopy

To explain the lubrication mechanisms, the steel/oil/electrolyte interface was characterized by electrochemical impedance spectroscopy (EIS) [6]. This technique is typically used to assess the anticorrosive properties of organic coatings. In the present case, EIS was used to study the behavior of release agents, with the aim of quantifying their protective actions of the steel substrate.

4.1 Experimental conditions

The mild steel S235 (10 × 10 cm) was used as working electrode. The steel was first polished to remove calamine. The oil was applied using a squeegee to obtain a homogeneous film. Film thickness, obtained by weighing, was about 1.5 µm. The cell was built by fixing a cylindrical Plexiglas tube ($S = 24\ cm^2$) above the plate. The counter-electrode was a large platinum plate (Fig. 8). A saturated calomel electrode (SCE) was used as reference. The electrolyte was a solution of 0.5 M of KOH + 0.1 M

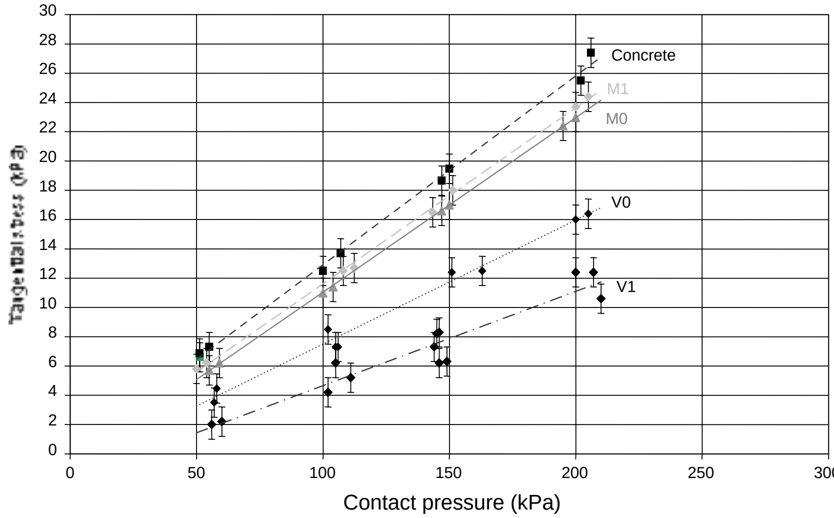


Fig. 7 Friction constraints versus contact pressure for pulverized and scraped films

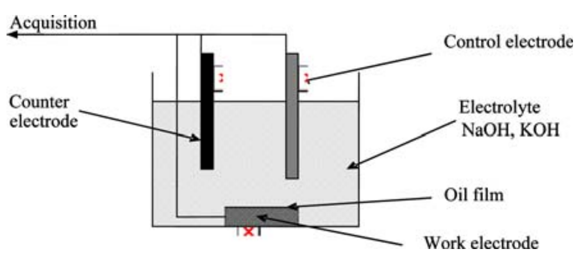


Fig. 8 Schematic representation of the electrochemical cell

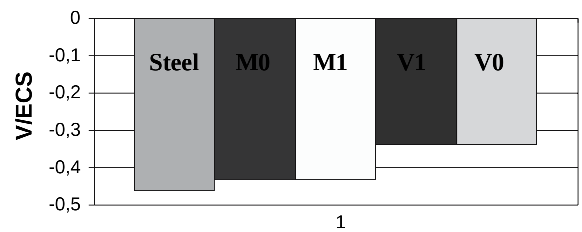


Fig. 9 Corrosion potential measured for each formulation after 1 h of immersion in the electrolyte

of NaOH. The pH of this medium was 13.2, similar to that of a concrete interstitial solution.

Electrochemical impedance measurements were carried out under potentiostatic regulation using a Solartron 1286 electrochemical interface with a Solartron 1250 frequency response analyzer (frequency range of 65 kHz to a few mHz). Each measurement was conducted on a new steel plate. The results were obtained from two experiments to ensure reproducibility. A measurement with the bare steel constitutes the reference.

4.2 Tests and results

Figure 9 compares the corrosion potential values obtained for each formulation. The corrosion potential is shifted towards more anodic potentials in the presence of oil films by comparison with the bare steel. With the vegetable oils (V_1 and V_0), this displacement is greater than with the mineral ones.

This first result suggests that vegetable oils provide better protection than mineral oils.

The impedance diagrams measured at the corrosion potential after 1 h of immersion in the electrolyte, for the mineral and vegetable formulations, are shown respectively in Fig. 10 and 11. Independently of the studied system, the diagram only shows one capacitive loop (one time constant).

The size of the capacitive loop is always smaller for the bare steel. Further, it is noteworthy that the oil layer applied on the metal surface does not give rise to the presence of a high-frequency (HF) capacitive loop on the diagrams. This type of HF loop is typically observed with paints, and characterizes the resistance of the electrolyte in the pores of the organic film. The tested oils were probably highly porous, and only reduced the active surface of the steel.

According to the appearance of the diagrams, the equivalent electrical circuit shown in Fig. 12 was used to obtain parameters characteristic of the interface. The slope of the curve log Z = f(log f) is slightly less than 1, which justifies the use of a constant phase element

Fig. 10 EIS diagrams at the corrosion potential after 1 h of immersion in the electrolyte (mineral films)

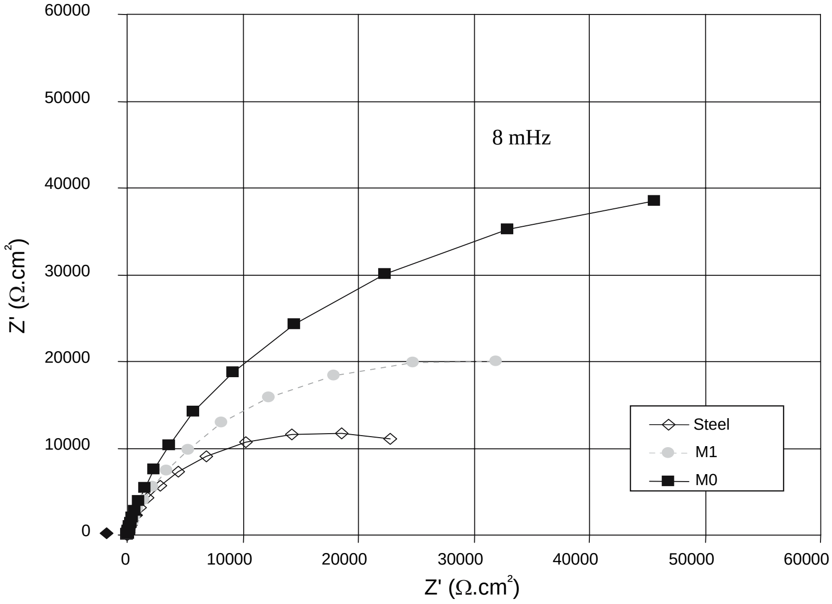
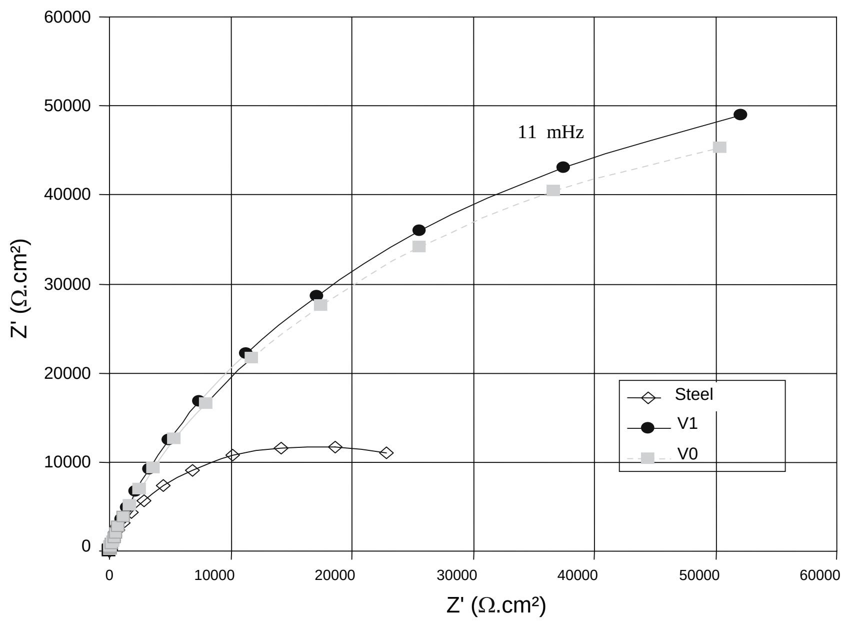


Fig. 11 EIS diagrams at the corrosion potential after 1 h of immersion in the electrolyte (vegetable films)



(CPE) instead of a capacity in the equivalent electrical circuit. The CPE impedance is given by

$$Z_{CPE} = \frac{1}{Q(j\omega)^{\alpha}} \text{ with } Q \text{ in } \Omega^{-1}\text{m}^{-2}\text{s}^{\alpha} \quad (1)$$

The film capacity was then calculated with the following equation [7]:

$$C = Q(Re^{-1} + R^{-1})^{(\alpha-1)/\alpha} \quad \text{C is expressed in Farads} \quad (2)$$

The values of R (Ω cm^2) and C (μF cm^{-2}) are reported in Table 4. The vegetable formulations present resistance values higher than those of the

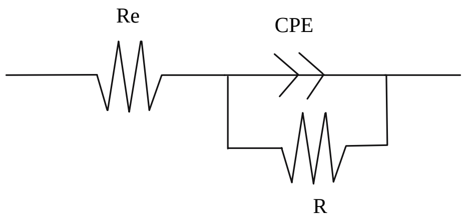


Fig. 12 Equivalent electrical circuit used to fit the experimental diagrams. Re: electrolyte resistance; R: polarization resistance; CPE: constant phase element (Q and α are the CPE parameters)

Table 4 Values of resistance and capacity for earch formulation

	R (Ω cm^2)	C (μF cm^{-2})
Steel	29300	443
M0	90500	269
M1	48700	362
V0	117000	261
V1	120000	247

mineral formulations. Conversely, the capacity values are lower with the vegetable formulations. These two observations reflect the fact that the vegetable formulations are more protective towards the surface.

In the mineral-oil category, M0 gives the best protection against corrosion, with a resistance of about 91000 Ω cm^2. With M1, the resistance drops further to 48700 Ω cm^2.

Oils V0 and V1 have resistance values of roughly 120000 Ω cm^2 which means the vegetable oils block are more efficient to block the anodic and cathodic sites of the steel.

The histograms in Fig. 13 represent the polarization resistance as a function of the oils and the acid value.

Without considering the use of an additive, the basic ester V0 is more effective against corrosion than the mineral base M0. This may be explained by the polarity of the ester molecules which is organized at the surface to form a continuous macroscopic film observed by optical microscopy. The addition of an acidifier leads to a significant decrease of resistance for mineral oils (M1) but tends to increase it for the vegetable formulation V1. Finally, a change of acid value has two different effects on the protective properties of films, according to the type of formulations.

5 Interpretation of results

5.1 Study of soap formation

To understand the role of acidifier at the interface, tests simulating the composition of the substances present at the interface have been performed.

The media “oil + interstitial cement solutions” were prepared according to the following protocol:

100 g of cement and 500 g of water were mixed for 3 min to get the interstitial solution. The solution is filtered to obtain a basic aqueous phase charged with calcium ions. One centimeter of oil was then added to the interstitial solution. The blend was shaken for 15 s, then left to rest for 1 h.

The structure of the media has then been observed (Figs. 14 and15).

M0: 2 phases (oil and water) without soap formation.
M1: 2 phases (oil and soap and water) with high amount of soap
V0: 3 phases (oil–soap–water) with low amount of soap
V1: 2 phases (oil–soap–water) with high formation of soap

Soap formation in V0 is limited. The presence of the acidifier increases the quantity of soap formed in V1. This result confirms that the acidifier acts as a catalyser of the saponification between a part of ester and the calcium hydroxides.

Moreover, the soap formed in M1 forms an emulsion with the oil, whereas with V1, the soap migrates to the surface of the oil. Consequently, it can be assumed that the soap forms micelles with the hydrocarbons leading to a stable emulsion.

According to the critical micellar concentration (CMC) of a calcium soap, the micelle formation is effective. The values of CMC in water and in ethanol are very low : CMCCa$^{2+}\sim$3,65.10^{-3} mol/L in ethanol determined by conductimetry.

The stages of soap micelle formation in an ester-water medium were described by G. Poulenat [8]. First, hydroxide ions diffuse towards the water-oil interface, where fatty acid salts form. Direct micelles form as soon as the concentration of fatty acid salts reaches the CMC. These micelles stabilize the water-oil interface and promote the formation of fatty acid salts.

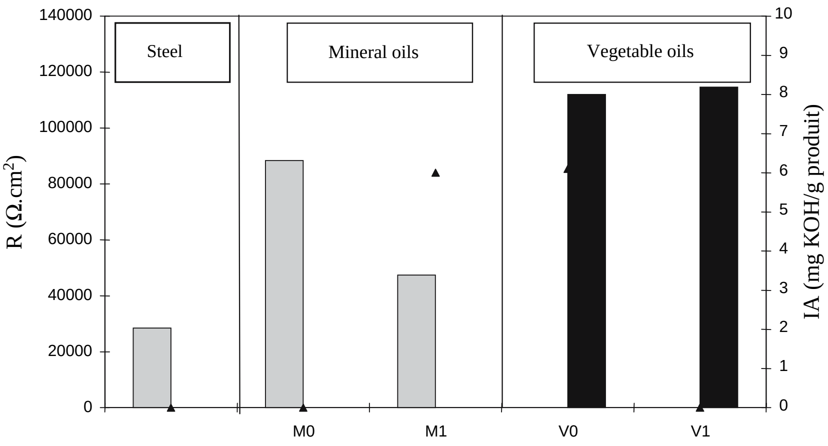


Fig. 13 Correlation between resistance of polarization and acid value

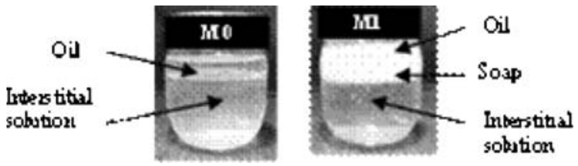


Fig. 14 Blends "cement solution + mineral oil", after 1 h rest

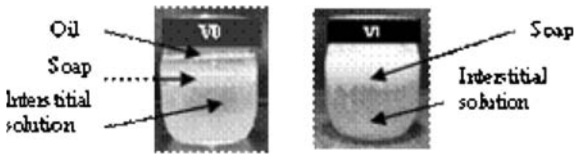


Fig. 15 Blends "cement solution + esters", after 1 h rest

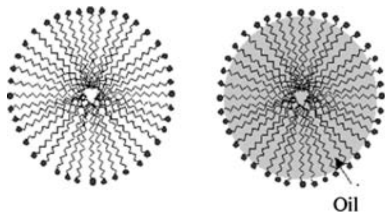


Fig. 16 Formation of micelle stabilized by a lipophile compound (oil)

Each micelle then behaves like a cell that brings into contact the oil and the calcium salt (Fig. 16).

Each micelle is surrounded by calcium ions and rejects one another ensuring their dispersion in the aqueous phase.

Finally, the soap behaves like a detergent, and tends to disperse the hydrocarbon molecules thus removing the film from its substrate.

5.2 Interface mechanisms

5.2.1 Mineral oils

Figure 17 schematizes the role of the mineral release agent. We assume that its mode of action involves concrete hydrophobing controlled by film thickness. This configuration minimizes friction by ensuring that the fines slip at the surface of the mould. Mineral oils are highly hydrophobic and thus promote

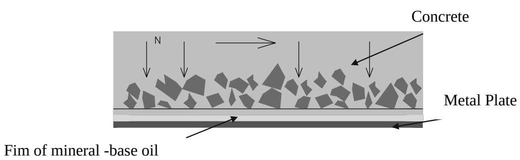


Fig. 17 Schematic representation of the influence of the mineral oil film at the concrete/formwork interface

concrete slippage in formwork thanks to the barrier effect created between concrete and formwork.

Therefore, the performances of lubrication are directly proportional to the thickness of the film.

According to this description, mineral oils act as a hydrophobic "barrier" against the concrete. Under these conditions, the acidifier does not take part to the friction reduction and penalizes the film resistance measured by EIS.

These results can be explained by the formation of micelles between carboxylates and hydrocarbons molecules at the mould-concrete interface [9] (Fig. 18).

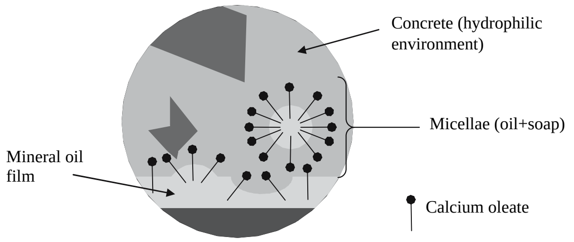


Fig. 18 Solubilization of the mineral film by the soap micelle

Calcium soap, which has low solubility in water, thus acts as a detergent [10] liable to solubilize the film. In this case, the steel substrate is less effectively protected. Alteration of the film generates reduction in impedances and causes the tribometric graphs to evolve.

5.2.2 Vegetable oils

The description of the mechanisms is based on the results obtained both by tribometry and by electrochemical impedance spectroscopy.

Without acidifier, the increase in the thickness of the vegetable oil film causes a reduction in friction stresses, reflecting a physical effect. The soap formation is limited, therefore the chemical effect is not perceptible.

The presence of the acidifier in V1 catalyses the formation of soap [8]. Then, the chemical effect takes precedence over the physical effect.

The surface tension L/V of soaps is representative of the tensioactive properties of soaps.

The measurements provides the following values : $\gamma_{L/V}$ oil + soap = 46 mN/m for $\gamma_{L/V}$ oil V1 = 36.1 mN/m. The calcium soap which increases the surface tension of the ester, has then a reduced catalytic effect which explains the saponification is not total [8].

We can assume that the interface organizes itself according to two layers [11] (Fig. 19). The ester molecules, owing to their polarity, adsorb preferentially to the metal wall of the formwork according to chemical interactions [12]. The calcium carboxylates orient themselves towards the hydrophilic concrete.

With V1, the association between the two effects (chemical and physical) seems to promote a significant reduction in friction stresses.

6 General conclusion

The comparison of the results obtained with the two techniques allows to explain the performances of

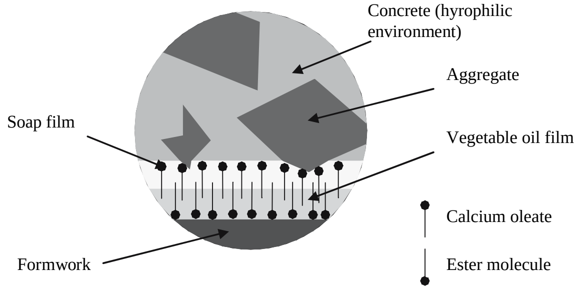


Fig. 19 Organisation of a soap film at the concrete/formwork interface

released agents according to the type of the base oil and the application. The two selected criteria, friction level and resistance to charge transfer, revealed good parameters to quantify the efficiency of the formulations. It was thus possible to identify the processes which control the reduction friction at the concrete-formwork interface.

The overall results allowed to build two models of the organization at the mould-concrete interface, due to the role of the calcium carboxylates (soap) which differs with the type of base oil.

The vegetable formulation showed a reduction in friction (46%) and a high resistance to charge transfer. These properties may be explained by a greater soap formation and by a specific interface organization of esters and fatty acid molecules in layers.

In the case of mineral oils, a hydrophobic physical film prevents the concrete from adhering to the formwork, generating a "barrier" effect controlled by film thickness. In this case, the rate of acidifier must be limited to avoid the alteration of the film.

Eventually, the chemical effect is the most efficient way to obtain a reduction in friction while controlling the thickness of the film applied to the formwork surface.

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