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Influence of the emulsions to the interface formwork/concrete

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Synopsis: The concrete is the ultimate building materials today and answers all the requirements of the modern architecture. The emulsion or the release agents are used to facilitate the concrete demoulding and to protect the formwork surfaces against corrosion. The final quality of the facings depends on the physicochemical characteristics of the demoulding products used. They must be selected according to the nature of the formwork and their compatibility with the casing skins. They must be consistently applied to the unit of the formwork, to a clean surface, in thin layers, uniform thickness. This agent must allow a placement of the more effective concrete while adhering perfectly to the formwork. The adherence of oil with the support is thus very important. It is studied by the determination of the adhesion energy Solid/Liquid on a formwork surface. This study is interested more particularly in the emulsions. Two forms are distinguished: Mineral reverses emulsion with the dispersed phase is water and Vegetable direct emulsion with the dispersed phase is oil. The first physicochemical results show an influence of the temperature on the adhesion energy. The quality of the concrete facings demoulded with emulsions appears better qualities that those carried out with oils demoulding.

Keywords: Emulsion, concrete, formwork, interface, adhesion energy, facing aspect, image analysis

Influence of the emulsions to the interface formwork/concrete

Laurent Libessart is an Associate Professor in the Department of Civil Engineering at the University of Artois, Béthune, France. His research interest includes of concrete/formwork interface, characterization of release agents and composite materials.

Amine Lahlou is a Graduate Student in the Department of Civil Engineering at Polytechnic school of Lille.

Chafika Djelal is a Professor in the Department of Civil Engineering at the University of Artois, Béthune, France. She is a member of RILEM committee 266-MRP (Measuring Rheological Properties of Cement-based Materials). She is the head of the division *geo-materials* of the laboratory (LGCgE). Her research interests include of composite materials, self-consolidating concrete, concrete/formwork interface, tribology and rheology of cement based materials and reinforcement of concrete structures.

Yannick Vanhove is a Professor in the Department of Civil Engineering at the University of Artois, Béthune, France. He is a member of RILEM committee 266-MRP (Measuring Rheological Properties of Cement-based Materials). His research interests include of self-consolidating concrete, form pressure generated by fresh concrete, tribology and rheology of cement based materials.

Sandrine Mateo is the aesthetic research team leader in the R&D CHRYSO department.

INTRODUCTION

Owing to its flexible setting conditions, concrete remains the most widely used material in the construction industry. The appearance of new concretes, coupled with the more demanding specifications imposed by project managers, has led to the marketing of increasingly efficient products (admixtures, demolding agents, etc.). However, the formwork removal step may give rise to certain problems, typically caused by an insufficient understanding of the mechanisms occurring at the concrete/formwork interface. In an effort to satisfy industry requirements as well as environmental protection and user safety conditions, many such products are plant-based. Among the array of demolding agents available on the market, emulsions meet the criteria for successful material placement due to their formwork removal attributes, in conjunction with cladding quality, which often raises conflict between project partners when works are submitted for acceptance. Over the past few years, this requirement has become a major economic challenge for the industry, yet emulsion use still tends to be limited. Moreover, the behavior of emulsions, whether plant-or mineral-based, has only received scant attention, especially as regards stability over time. This lack of investigation is in sharp contrast with the case of demolding oils, which have been extensively studied [1, 2], as evidenced in the bibliography. The emulsions make it possible to apply less oil to the formwork surface and thus to reduce the total quantity of product. It is important to control this application to ensure a good demolding. The suppliers advise the pulverization followed or not of a scraping or a brushing. After the application, latency is advised of approximately 15min to ensure a total evaporation of the absorbent phase.

As regards demolding agent classification, the SYNAD (French Association for Concrete and Mortar Admixtures) created an abacus [3] based on product composition as well as a set of criteria related to its use (fire safety, health, environmental considerations).

Concrete cladding is generally visible once the structure has been built; it forms the structural skin, which must resist the range of stresses acting on the structure throughout its service life and blend in with its environment as naturally as possible. Achieving high-quality cladding necessitates considerable attention in: designing the concrete mix, establishing component homogeneity, and laying out the various setting steps. The quality of formwork and demolding product, plus the casting sequence and use of vibration, all contribute to ensuring a satisfactory final result.

The goal of this study is to correlate the physicochemical properties of mineral-and plant-based emulsions with cladding appearance after formwork removal, as a means of improving understanding of the concrete/formwork interface. The main parameters analyzed are: type of emulsion and influence of both temperature and wait time prior to concrete casting.

EXPERIMENTAL INVESTIGATION

This section discusses the materials and physicochemical characterization techniques used to study emulsions. Also detailed here in will be the procedure for analyzing concrete cladding images.

Materials

Release agents

The demolding agents selected for this study are both mineral and plant emulsions.

Two emulsion forms are distinguished (**Fig. 1**) [4]: a mineral-based inverse emulsion with water as the dispersed phase; and a direct plant-based emulsion with oil as the dispersed phase.

From a practical perspective, the majority of emulsions are composed of an aqueous liquid phase and a phase with limited water solubility.

In oily emulsions, the hydrophobic part of the emulsifier molecule is found in the dispersant phase (oil) and, conversely, in the other type of emulsion (as shown in **Fig. 1**). Emulsion formation relies heavily on a stable environment. To prepare an emulsion, the oily and aqueous phases need to be assembled and stirred together, along with the addition of one or more emulsifying agents. The efficiency of this emulsification process depends on both the stirring mode and intensity. The emulsifier must also be introduced under appropriate conditions; it must feature a structure capable of strengthening interphase linkage to the greatest extent possible. Such is a key element in ensuring mix stability [6].

Table 1 lists the various characteristics provided by the manufacturer.

At construction project sites, the demolding agent is most often sprayed onto the formwork or spread after spraying with a rubber scraper in order to remove excess product relative to each single layer. For this experimental campaign, only the spray-on application will be studied.

Concrete

A CEMI 52.5 CP2 type of Portland cement was used for this investigation, while the concrete employed to study cladding aesthetics was of a conventional variety containing 28% cement paste. The workability, as measured by the Abrams cone test, must lie between 12 and 14 cm. The specific mix design is given in **Table 2**.

Formwork

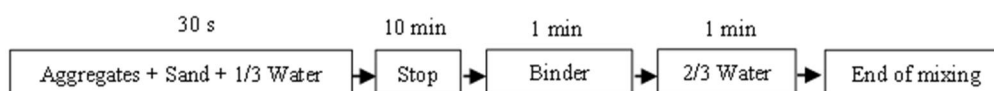
Metal formwork is extensively introduced in the building trades; it serves to streamline concrete setting and enhances worksite productivity. Formwork reuse possibilities are substantial for structural elements repeated throughout a project, i.e.: vertical shear walls, floors, and columns. The use of stiffeners enables extending elements over large surface areas.

Before characterizing the wettability and determining the bonding energy of the various mix designs, the surface roughness was first measured.

This new formwork surface used [7] was homogeneous and devoid of any defects or particularities regarding its lamination. Its roughness was measured at $1.27 \mu\text{m}$ [8].

Mixing protocol

Mixing was carried out in accordance with Standard NF P 18-404, entitled "Concretes: Mix design, suitability and inspection tests - Casting and storage of test specimens", which specifies the following operating procedure:



Tests were conducted in the laboratory at room temperature (around 20°C).

The cladding analysis relies on molds measuring $30 \times 30 \times 30 \text{ cm}^3$. The concrete is cast in three layers and then vibrated at 5 points for 10 seconds per layer using a 40-mm diameter vibrating needle. The vibration step is carried out in five 10-second sequences per layer with the 40-mm needle. All emulsions studied were applied by spraying, with a wait time of 15 min prior to concrete casting. This application was completed without experiencing any leakage or overload.

Methods for characterizing the physical chemistry of emulsions

Rheological properties

The rheological properties of demolding oils exert a powerful influence on the behavior of the concrete/oil/formwork interface. This influence is directly felt on: wettability, friction stresses at the concrete/formwork interface, and ultimate cladding quality [9, 10]. The viscosity of the two oils is determined using a HAAKE VT550 single-speed viscometer, fitted with a 1° (PK5) cone. A constant volume of oil ($v = 0.6 \text{ cm}^3$) is deposited on the plate with a pipette, and the cone is then lowered toward the plate. To obtain an accurate measurement, a 5- to 10-min wait time (depending on measurement temperature and after depositing the oil on the plate) is required for the oil to reach the desired temperature. During the testing phase, temperature must be held constant so as not to bias the reading. Viscosity measurements were conducted at three constant temperatures (10°C , 20°C and 40°C).

Surface tension

The liquid/vapor surface tension of a liquid is correlated with the value of the contact angle formed by a drop on a support [11]. Contact angles are a readily observable physical manifestation of the more fundamental concepts of energy and surface tension. This insight serves to determine the properties of wettability and adhesion of a film, which in turn makes it possible to predict the properties of a lining or detect the traces of surface pollutants. As part of a theoretical approach, adhesion energy values are calculated based on contact angle data. It may be considered that the liquid/solid adhesion energy value is representative of the adhesive strength between the liquid and the support.

Young [12] was the first researcher to derive an equation for the equilibrium of a drop of liquid placed on a given solid. This equation is expressed in the following form:

$$\gamma_{LV} \cos(\theta) = \gamma_{SV} - \gamma_{SL} \Leftrightarrow \gamma_{SL} = \gamma_{SV} - \gamma_{LV} \cos(\theta) \quad (1)$$

where θ is the contact angle formed by the surface and the plane tangent to the meniscus, γ_{SV} the solid surface energy, γ_{LV} the liquid/vapor surface tension et γ_{LS} the liquid/solid interfacial tension.

The liquid/solid adhesion energy value is also representative of the adhesive force between a liquid and its support. Adhesion energies are calculated according to Zisman's Equation [13]:

$$\omega_{LS} = \gamma_{LV} \times (1 + \cos \theta) \quad (2)$$

In order to determine the solid/liquid adhesion energy, it is necessary to measure both the contact angle of the oil and the liquid/vapor surface tension.

The liquid/vapor surface tension was measured using Wilhelmy's plate method at 20°C. The device employed is a tension meter manufactured by the CAD firm (**Fig. 2**) fitted with a platinum blade. According to this method, a thin platinum plate is hooked to a Kahn scale and then immersed into a liquid contained in a tank. The surface tension is indicated by the meniscus formed along the plate boundary.

The material contact angle was determined using a PGX type of mobile goniometer. This device measures the contact angle at the interface between a liquid drop and the substrate surface where it has been deposited.

EXPERIMENTAL RESULTS AND DISCUSSION

Dynamics viscosities

Table 3 presents the dynamic viscosities obtained by averaging the results from 3 tests. Given its low viscosity value, the EDV2 reference could not be measured. Results show that the demolding agents display a linear behavior without any yield point: the behavior of emulsions is thus Newtonian [10].

Viscosity decreases as a function of temperature, hence making it important to control the film application after spraying.

Liquid/vapor surface tension

The L/V surface tension values, derived at 10°, 20° and 40°C, are given in **Figure 3**. These findings suggest that increasing the temperature causes the surface tension to drop. Since the molecules are becoming more thermally excited, they are more likely to leave the surface and move toward the interior. The force attracting molecules toward the interior should therefore decrease as temperature rises. This analysis demonstrates that film adhesion may be modified depending on temperature. Nonetheless, values remain in the vicinity of 30 mN/m, as recommended during previous studies [8]. For the remainder of this study, results will be exclusively analyzed at 20°C.

Wettability

Instantaneously

The drop angles, provided in **Table 4**, all lie between 20° and 35°, with the inverse emulsions yielding the smallest angles. Emulsions must therefore stabilize over time, and it is preferable to track the evolution in wettability over time as well.

Over time

Measurements spanning a time period were carried out in order to study the variation in demolding agent wettability. **Figure 4** shows the evolution in wettability vs. time for the four tested emulsions. Wettability

stabilizes after some 7 min for EDV1 and at 5 min for EDV2 and EIM1. These values all lie between 10° and 20°. It is also observed that prior to stabilizing the drop, a significant slope suddenly appears for both emulsions. This variation may be due to many parameters (e.g. type of emulsion, emulsion mix design, water evaporation). The surface tension balance becomes modified at the formwork surface. The plate roughness parameter might also serve to influence this variation. Water evaporation leads to greater emulsion wettability on the support as the drop spreads more readily.

Adhesion energy

Instantaneously

Figure 5 summarizes the various adhesion energy values at $t = 0$ min and at 20°C. Emulsions EDV1, EIM1 and EIM2 all display a satisfactory adhesion energy level of at least 70 mN/m. Only the EDV2 value lies below this threshold. It can thus be concluded that demolding agents must exhibit the highest possible surface tension and the lowest drop angle.

Over time

This change in wettability thus implies a change in adhesion energy between $t = 0$ and $t = 20$ min. **Figure 6** depicts such an evolution over time. The rise in adhesion energy values reveals the benefit of allowing the water contained in the emulsion to evaporate as a means of generating the best possible adhesion. It is preferable to let the evaporation step last at least 5min after emulsion application in order to ensure that all the water has evaporated and a homogeneous oil film has been obtained.

Analysis of concrete cladding images

The most significant cladding defect is a color variation, which produces a visual differentiation between large adjacent surface zones [14, 15]. This kind of alteration typically affects concrete over several millimeters of thickness. Two types of color variation exist, namely those between different concrete mixes on a single cladding, and those on the same mix. A rating scale with 7 shades of gray has been developed by CIB [16] (**Fig. 7**). French Standard P18-503 also offers a guideline for evaluating the surface appearance of concrete cladding [17]. This standard is based on three characterization criteria: flatness, texture (bubbling), and hue. To analyze cladding and formwork surfaces, the rating criteria selected were those used by our team when testing for the influence of mix designs on cladding quality. For this cladding, a rating is determined 24hours after formwork removal. The cladding appearance, microbubbling, dusting, fouling, shading and snag points are all parameters capable of characterizing formwork removal quality [7].

The color variation analysis entails an image analysis. The cladding obtained was photographed on days 1, 3 and 7 following formwork removal. The concrete blocks were released 12hours after casting and then stored indoors at a temperature of 20° +/- 2°C and at a constant humidity of 60% +/- 5%.

The first measurement stage consists of a general assessment of the cladding, complete with snapshots of its overall appearance.

To perform this assessment, the following procedures are required:

- Specify the average color through use of the color chart [16].
- Eliminate any artifacts attached to surface edges.
- Calculate the total cladding surface area.
- Determine the rate of color variation.

Two freeware applications are available for this step: GIMP 2.8, and ImageJ 1.48.

The types of cladding obtained with EDV1 and EIM1 are shown in **Figure 8**.

At first, the photograph is adjusted to yield an image without any background capable of influencing the image analysis. Next, the color, brightness and saturation are all improved, followed by a binarization operation involving three's holding. For the color variation, it is critical to select shade-of-gray 1 or 2 from **Figure 3** as the reference. This scale is then compared with the photograph taken of the concrete cladding. To determine the color variation rate value, an appropriate measurement scale needs to be defined. As a final step, the "black" zones are calculated. **Table 5** lists the full set of color variation results for the demolding agents studied herein.

No notable differences appear between the direct and inverse emulsions, nor in the selection of mineral-based vs. plant-based products, with very good cladding quality measured at 7 days.

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The visual analysis reveals little fouling and few snag points; moreover, no dusting was detected on the cladding. Microbubbling was found to be relatively sparse (approx. 0.25%), which indicates that the emulsion composition does not prevent air bubbles from rising to the surface.

Figures 9 and 10 present the color variation for cladding obtained with emulsions EDV1 and EIM1. The black zones show the variation in color created with respect to the reference color chosen on the CIB scale. No shade of gray is identified, as all zones with a higher color value have been darkened. For purposes of this study, level 1 was selected. Since bubbling may slightly alter the values, it needed to be removed from the calculation.

CONCLUSIONS

The emulsions studied as part of this research have demonstrated strong physicochemical performances. Their viscosity values are lower due to the presence of water in their composition, hence making it possible to apply these emulsions using an adapted spraying protocol between 10°C and 40°C. The drop angles lie near 25° (especially for plant-based emulsions), while their liquid/vapor surface tension is close to 30 mN/m, thus providing for a good level of adhesion energy. The change in wettability over time serves to homogenize the surface of the film being applied; resulting in a greater bond with the surface, which further increases the adhesion energy value. It is thus essential to wait 5 - 10 minutes before setting the concrete into the formwork. Cladding quality improves over time; consequently, it is preferable to wait at least 7 days to derive an evaluation. While the color does visibly evolve, a homogeneous color pattern can be achieved after 7 days. It is also necessary to develop the images analysis on a microscopic scale (porosity analyses) and by spectrophotometry measurements. A complementary study can be realized to know the emulsion influence on the concrete friction on the formwork by tests with a plane/plane tribometer. The analysis of anti-corrosive protection can be given by Electrochemical Impedance Spectroscopy (EIS).

FURTHER RESEARCH

The continuation for the study will be to analyze the various types of application and to carry out an experimental campaign on a plane/plane tribometer in order to release the coefficients of friction of the concrete on the various formulations and thus to supplement the physicochemical study.

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REFERENCES

- [1] Djelal C., De Caro P., Libessart L., Dubois I., Pébère N., "Comprehension of demoulding mechanics at the formwork/oil/concrete interface", *Materials and Structure*, V. 41, No. 3, 2008, pp. 571-581.
- [2] Hermann K., "Produits de décoffrage", *Bulletin du ciment*, n° 15, 1993.
- [3] Syndicat national des adjuvants pour bétons et mortiers (SYNAD), *Classification Synad des agents de démoulage*; 2014.
- [4] Zolfaghari R., Fakhruddin Razi A., Abdullah L.C., Elnashaie S., Pendashteh A., "Demulsification techniques of water-in-oil and oil-in-water emulsions in petroleum industry", *Separation and Purification Technology*, V. 170, 2016, pp. 377-407.
- [5] Doumeix O., "Opérations unitaires en génie biologique - Tome 1 - Les émulsions", 2011, p.116
- [6] Fingas M., Fieldhouse B., "Studies of the formation process of water-in-oil emulsions", *Marine Pollution Bulletin*, V. 47, 2003, pp. 369-396.
- [7] Libessart L., Djelal C., De Caro P., "Influence of the type of release oil on steel formwork corrosion and facing aesthetics", *Construction & Building Materials*, V. 68, 2014, pp. 391-401.
- [8] Libessart L., de Caro P., Djelal C., Dubois I., "Correlation between adhesion energy of release agents on the formwork and demoulding performances", *Construction & Building Materials*, V. 76, 2015, pp. 130-139.
- [9] Hart I., "Concrete release agent, associated materials and surface finishes", *Concrete*, V. 28, No. 12, 1994, pp. 33-34.
- [10] De Caro P., Djelal C., Libessart L., Dubois I., "Influence of the nature of demoulding agent on the properties of the formwork/concrete interface", *Magazine of Concrete Research*, V. 59, No. 2, 2007, pp. 141-149.
- [11] Chappuis, J.; Georges, J-M. "Contribution to Study of Wetting - Analysis of a Measuring", *Method. J. Chim. Phys. Phys. Chim. Biol.*, V. 71, 1974, pp. 567-575.
- [12] De Gennes P.G. "Wetting: statics and dynamics", *Reviews of modern physics*, V. 57 No. 3, 1985, pp.827-863.

- [13] Zisman W.A., «Relation of the Equilibrium Contact angle to Liquid and Solid Constitution», *Advances in Chemistry*, V. 43, 1964, pp1-51.
- [14] Collection technique CimBéton. Bétons et ouvrages d'art 6 La maîtrise esthétique des parements en béton. T49; 2005.
- [15] Lemaire G, Escadeillas G, Ringot E. «Evaluating concrete surfaces using an image analysis process», *Construction & Building Materials*, V. 19, 2005, pp. 604611.
- [16] CIB, « Tolérances sur les défauts d'aspect du béton », Rapport n° 24 6 n°1189, 1973.
- [17] ST P18-503, «Concrete surfaces and facing. Identification elements», ISSN:0335-3931, 1989.

TABLES AND FIGURES

Table 16 Release agents properties

	EDV1	EDV2	EIM1	EIM2
Color	White	White	White	White
Nature	Mineral	Mineral	Vegetable	Vegetable
Flash point, °C	> 100	> 100	85	96
Density	< 1	0.985 ± 0.02	< 1	< 1

Table 26 Concrete formulation

Concrete	B28
Paste volume (%)	28
Cement CEM I 52,5 CP2 (kg/m ³)	232
Fillers (kg/m ³)	77
Sand 0/4 (kg/m ³)	838
Gravel 4/8 (kg/m ³)	287
Gravel 6/20 (kg/m ³)	778
Water (l)	176
W/B	0.57

Table 36 Dynamic viscosity of emulsions

	Viscosity at 10°C (mPa.s)	Viscosity at 20°C (mPa.s)	Viscosity at 40°C (mPa.s)
EDV1	43.5 ± 0.7%	38.9 ± 7.1%	27.9 ± 1%
EDV2	6	6	6
EIM1	62.8 ± 0.63 %	53.4 ± 7%	31.8 ± 6.7%
EIM2	79.1 ± 8.9%	60.6 ± 2.9 %	34.9 ± 2%

6 = not measured items.

Table 46 Instantaneous contact angle of emulsions at 20°C

	Contact angle (°)
EDV1	33.3
EDV2	35.1
EIM1	28.2
EIM2	23.1

Table 56 Percentage of color variation on days 1, 3 and 7

	1 day	3 days	7 days
EDV1	9.75	4.25	3.5
EDV2	13.45	5.11	2.65
EIM1	13.71	3.64	2.94
EIM2	7.23	5.43	3.31

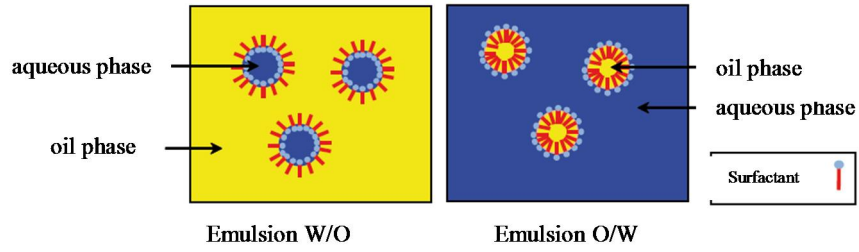


Fig. 16 Emulsion forms [5]



Fig. 26 CAD tensiometer

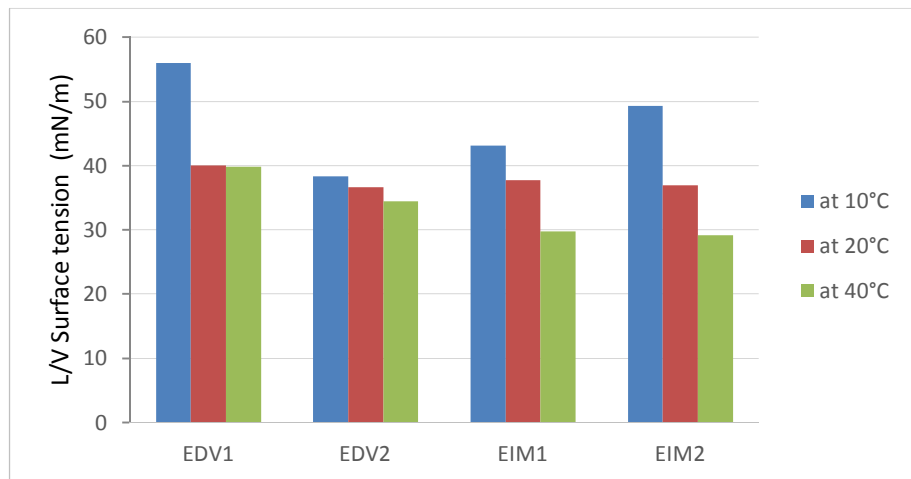


Fig. 36 Liquid/vapor surface tension (10°C, 20°C and 40 °C)

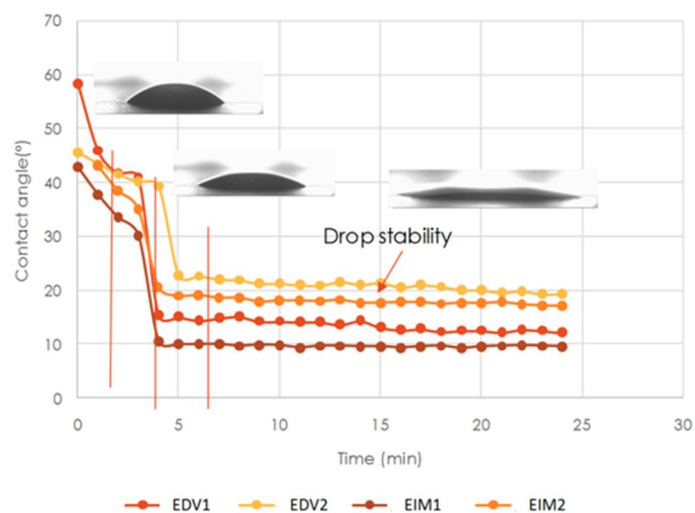


Fig. 46 Evolution of the drop angles in time

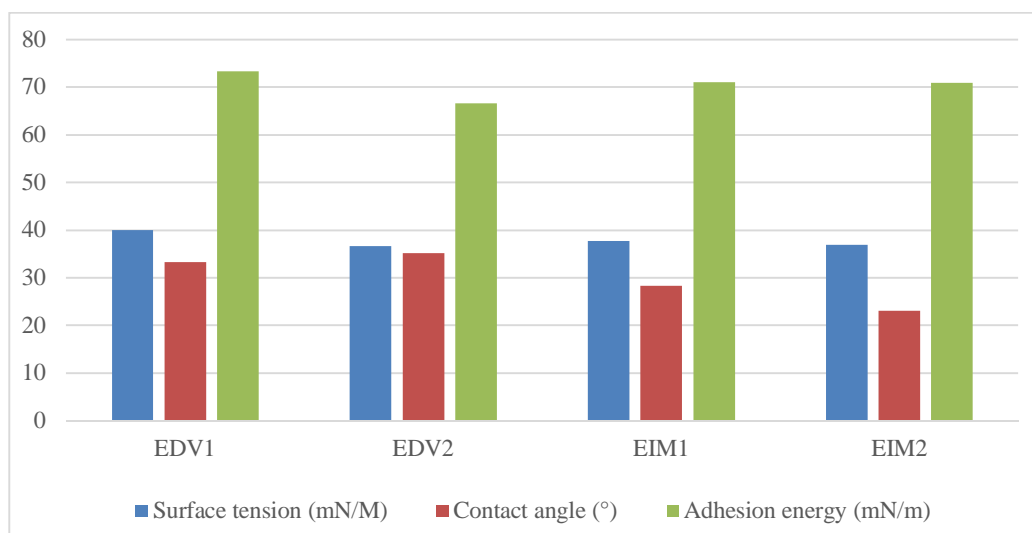


Fig. 56 Instantaneous adhesion energies of the emulsions

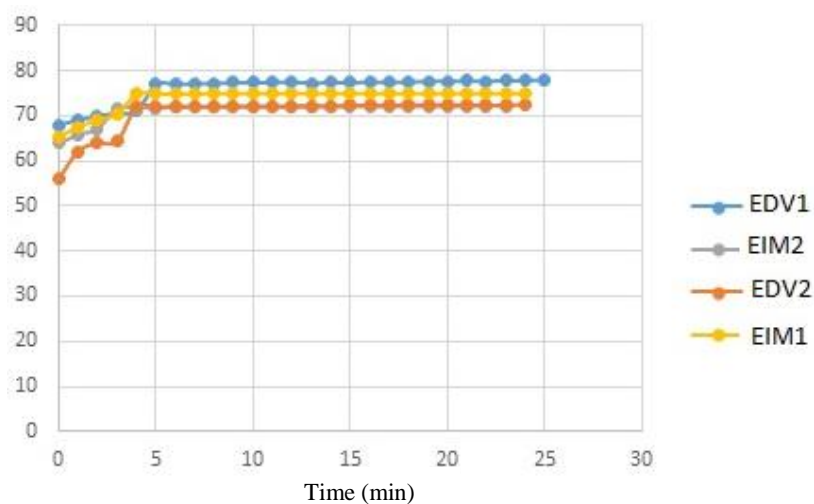


Fig. 66 Over time evolution of adhesion energy

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Fig. 7 6 CIB shades of gray

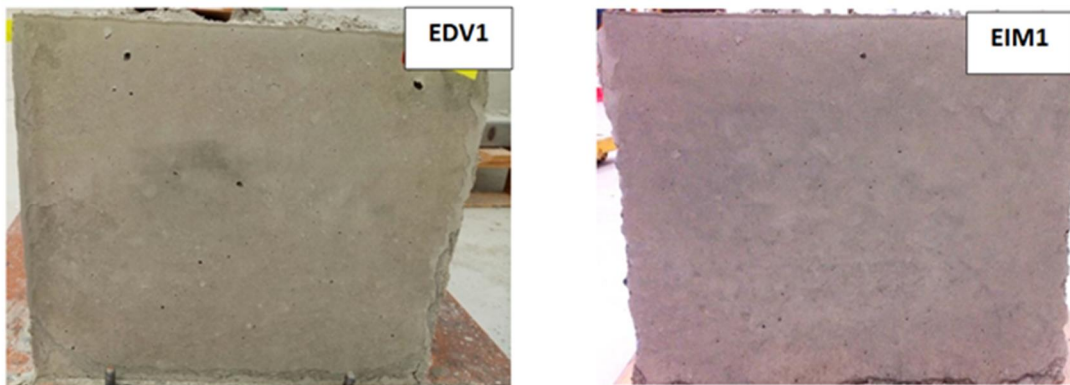


Fig. 8 6 Concretes cladding EDV1 and EIM1 on day 1

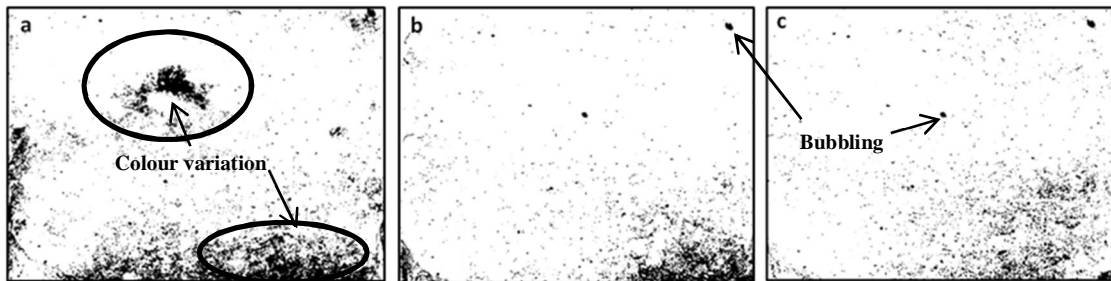


Fig. 9 6 Images analysis of EDV1 on days 1 (a), 3 (b) and 7 (c)

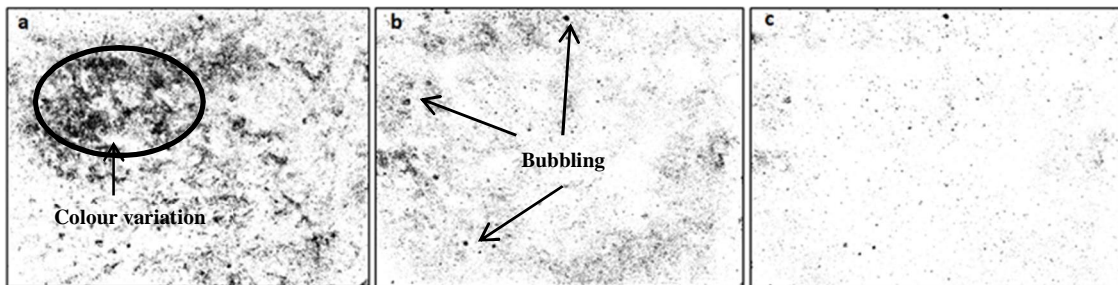


Fig. 10 6 Images analysis of EIM1 on days 1 (a), 3 (b) and 7 (c)