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Monitoring the early stiffness development in epoxy adhesives for structural strengthening

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ABSTRACT

The present work aimed to assess the early-age evolution of *E*-modulus of epoxy adhesives used for Fibre-Reinforced Polymer (FRP) strengthening applications. The study involved adapting an existing technique devised for continuous monitoring of concrete stiffness since casting, called EMM-ARM (Elasticity Modulus Measurement through Ambient Response Method) for evaluation of epoxy stiffness. Furthermore, monotonic tensile tests according to ISO standards and cyclic tensile tests were carried out at several ages. A comparison between the obtained results was performed in order to better understand the performance of the several techniques in the assessment of stiffness of epoxy resins. When compared to the other methodologies, the method for calculation of *E*-modulus recommended by ISO standard led to lower values, since in the considered strain interval, the adhesive had a non-linear stress–strain relationship. The EMM-ARM technique revealed its capability in clearly identifying the hardening kinetics of epoxy adhesives, measuring the material stiffness growth during the entire curing period. At very early ages the values of Young's modulus obtained with quasi-static tests were lower than the values collected by EMM-ARM, due to the fact that epoxy resin exhibited a significant visco-elastic behaviour.

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1. Introduction

In recent years, the application of thermosetting resins in civil engineering applications has largely increased, mainly for their use in structural strengthening systems such as Fibre-Reinforced Polymer (FRP) reinforcements [1]. The most common resins employed as structural adhesive for bonding FRP to structural elements to be strengthened are two-component epoxy resins [2,3].

In FRP installations, the mechanical behaviour of the strengthening system is strongly influenced by the epoxy adhesive, particularly at early ages, while the mechanical properties of the adhesive are still enduring significant evolution. Therefore, the final performance of the whole application strongly depends on adequate preparation, application and curing of the epoxy resin itself. During the curing period, the fluid resin transforms into a rubber (gelation) and then in a solid glass (vitrification), developing a progressively denser polymeric network [4].

It has been shown that the necessary curing time to reach the targeted bond strength of a given resin significantly depends on environmental conditions, such as temperature and moisture [5,6]. Lapique et al. [7] and Moussa et al. [8] have investigated the effect of the curing temperature on the development of the mechanical properties of epoxy resins generally utilized for structural strengthening, reaching similar conclusions: as both time and temperature increase, the epoxy tensile strength increases. Additionally, adhesives used for FRP strengthening applications must cure in-situ at various environmental conditions.

From the above considerations, it is clear that the development and implementation of non-destructive methods that are able to provide continuous information correlated to the curing process of epoxy resins are of paramount importance for in-situ applications of strengthening systems. The determination of the time at which the material becomes actually capable of bearing structurally relevant stresses is fundamental (and especially important in the case of pre-stressed systems [9]), as well as the stiffness increase along time. In fact, the elasticity modulus (*E*-modulus) of a polymer material such as cured epoxy adhesive is one of the most significant material parameters in structural analysis. Usually *E*-modulus values are obtained from tensile mechanical tests under a monotonic state of stress and there are already available several standards (e.g. ISO 527-1:2012 and ASTM D638M-93). It is nonetheless remarked that some of these standards are specifically directed to secant *E*-modulus, and do not take into account the strength of the material in the testing/calculation

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procedure. Additionally, tensile tests can solely provide measurements at discrete instants and they are complicated to perform on the construction site (most of the times due to economical unfeasibility).

For the aforementioned reasons, alternative vibration-based techniques are commonly utilized nowadays for the study of polymer-based plastic materials. The most widely used vibration-based techniques for *E*-modulus determination are the resonant frequency-based methods [10], as well as the dynamic mechanical analysis (DMA) [11]. Both these methods require the application of a mechanical force and therefore it is clear that these techniques are only suitable for specimens which have developed enough stiffness at the age of testing. Moreover, there are reports that the elastic modulus values obtained from DMA show large discrepancies between specimens and are in many cases different from those measured by quasi-static mechanical tests, as can be seen in the paper of Deng et al. [11]. Another promising dynamic test method is the novel torsion pendulum test developed by Yu et al. [12]. This technique allows measuring the dynamic shear modulus of adhesives since the early stages of the cure, monitoring the change of the resonant frequency with time.

Alternatively, techniques based on the propagation of ultrasonic waves through the sample can provide continuous measurement of the elasticity modulus of composite materials, ranging from the fluid-like stage up to the fully hardened state [13]. Even though these wave-based methods allow overcoming some of the drawbacks of the conventional techniques based on the resonance frequency, there are relatively frequent problems in the interpretation of the signals, and some of the basic assumptions for the interpretation of results (e.g. the tested material should be homogeneous and isotropic) are jeopardized when applied to a composite material as the case of epoxy resin [14].

Azenha et al. [15] proposed a novel method to measure *E*-modulus evolution in concrete, called Elasticity Modulus Measurement through Ambient Response Method (EMM-ARM). The method is based on the direct measurement of the evolution of the natural frequency of vibration of a composite beam, filled with the material under testing. The evolving natural frequency of the composite beam can thus be directly converted into the *E*-modulus of the tested material, based on the dynamic equations of motion of the system. The method allows continuous concrete *E*-modulus measurements immediately after casting and was applied also to identify the elastic modulus evolution of cement pastes, mortars and stabilized soils [16].

The present work aimed to study the early-age evolution of *E*-modulus of epoxy materials used for FRP applications, and better understand the relationship between distinct approaches for its assessment. For this purpose, a simultaneous study of *E*-modulus of the same adhesive mixture was carried out through EMM-ARM, together with tensile testing according to ISO standards (monotonic secant *E*-modulus) at several ages. Furthermore, since no publications were found in literature related to the intercomparison of the epoxy *E*-modulus by means of monotonic tensile tests (such as the case of the ISO standard) and cyclic tensile tests, specific experiments were performed in such concern. Overall, this research work assists clarifications about the applicability of several approaches/techniques in predicting the stiffness of epoxy resins.

2. Experimental programme

The experimental programme consisted in the execution of an epoxy resin mixture and the characterization of the corresponding stiffness evolution along the curing time by monotonic tensile tests (MTT), cyclic tensile tests (CTT) and EMM-ARM tests. The two-component epoxy resin-based adhesive used in the experimental work, produced by S&P® Clever Reinforcement, had the trademark 'S&P Resin 220 epoxy adhesive'. This adhesive is typically employed for bonding FRP laminates to concrete and steel, and therefore may be seen as representative. According to the manufacturer [17], the component A (resin) contains 20–25% (by weight) Bisphenol A-Epoxy Resin and 5–10% Neopentyl glycol diglycidyl ether and, the component B (hardener) includes 20–25% poly (oxypropylene) diamine, 1–2.5% piperazine and 20–25% 3,6-diazaoctanethylenediamin; triethylenetetramine. All the specimens tested in the scope of this research were originated from a single batch that involved a total volume of epoxy resin of ~1.2 l. The individual components were separately stirred and then component B was added to component A at a ratio of 1:4 by weight of the respective constituents. To minimize air inclusions, the compound was thoroughly and slowly manually mixed until the colour was uniformly grey and free of any streaks. The whole mixing procedure lasted approximately 4 min.

All experimental procedures (mixing and testing) took place under controlled environmental conditions (in climatic chamber), with temperature of 20 ± 1 °C and relative humidity of $55 \pm 5\%$. The following sections detail the programme of tests, methods and the procedures of test series, including sample geometries, test configurations and preparation of specimens.

2.1. Tensile tests – MTT and CTT

An extensive set of 30 tensile tests were performed in order to determine the epoxy *E*-modulus at several ages.

The specimens for testing were manufactured according to "type 1A" defined in EN ISO 527-2:2012. This specimen's geometry is characterized by having a dog bone shape at both extremities, with a thickness of 4 mm and overall geometry defined as shown in Fig. 1. Teflon moulds were devised for fabrication of the specimens. After mixing the two resin components, the homogenized compound was cast into the referred Teflon moulds. Afterwards an acetate sheet was placed on the top surface and pressed with a steel roller. The specimens were kept sealed in the curing environment and were removed from the moulds just before being tested. For all specimens, width and thickness were measured at the three sections (S1, S2 and S3) identified in Fig. 1, using a digital calliper with a precision of ± 0.01 mm, to check tolerances and for longitudinal stress calculation.

The experimental programme comprised the testing ages of 12, 18, 36 and 84 h. For each age of testing, three monotonic tests and three cyclic tests were carried out. It is however remarked that the mentioned cyclic tests were always performed on the same three specimens: i.e., after being tested at the age of 12 h, the specimens were stored in the curing environment and re-tested at 18 h, 36 h and 84 h. This strategy was adopted because of the low levels of stress induced during the cyclic testing (1/3 of the tensile strength at the age of testing), which should not induce any kind of damage to the specimen and allow re-using it. Nonetheless, in order to confirm the feasibility of this re-utilization of specimens, three additional specimens were cast for cyclic testing solely at the age of 84 h from a virgin state. The comparison of results of testing of the three virgin specimens and the three re-used specimens at the age of 84 h can assist the assessment of the influence of the

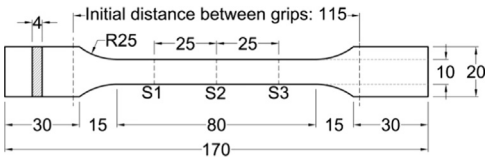

Fig. 1. Specimen dimensions according to ISO 527-2. Note: all dimensions are in millimetres.

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