



# High-temperature slurry erosion of vinylester matrix composites – The effect of test parameters

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## ABSTRACT

Glass fibre (GF) reinforced vinylester composites (VE-FRP) are commonly used materials in hydro-metallurgical reactors, the pulp and paper industry and waste water treatment plants, due to their excellent chemical resistance combined with good mechanical performance. In these applications, materials can be subjected to erosion, elevated temperatures (as high as 95 °C) and various chemical environments. However, studies on the slurry erosion of vinylester-based composites at high temperatures have not yet been reported.

In this study, the erosion resistance of GF reinforced VE-FRP was investigated with a pilot-scale reactor. The effect of slurry concentration, erodent particle kinetic energy and slurry temperature was studied. The dominating wear mechanism was found to be abrasive wear. The VE-FRP structure was found to be prone to erosive turbulent flow and cavitation. Moreover, an increase in the erodent concentration of the slurry (10–20 wt%) or in the total kinetic energy of the erodent particles (30–770 kJ) increased the wear rate of the material markedly (up to 6 times higher weight loss). However, the total effect of different interrelated parameters was found to be complex. Consequently, it is recommended that predictions of the erosion rate of VE-FRP components are based on tests carried out in conditions that simulate the actual service environment.

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## 1. Introduction

Glass fibre (GF) reinforced vinylester composites (VE-FRP) have excellent chemical resistance combined with good mechanical performance. Hence they are commonly used construction materials in the harsh chemical environments of the hydrometallurgical process industry, the pulp and paper industry and waste water treatment plants [1]. In the hydrometallurgical processing of metals, construction materials can be subjected to erosion, elevated temperatures (as high as 95 °C), and various chemical environments, like sulphuric acid [2]. Thus mastering the erosion properties of GF-reinforced VE-FRPs at high temperatures is essential in optimising the maintenance intervals of different FRP equipment. The avoidance of unnecessary process shutdowns would provide major operating cost savings. However, the slurry erosion behaviour of VE-FRPs has not been studied at elevated temperatures.

The complex composite structure consisting of matrix and reinforcement(s) and its anisotropic and viscoelastic nature make the study of the erosion behaviour of composites a challenge. The wear mechanisms that take place during polymer erosion depend highly on the polymer type. Brittle polymers are susceptible to crack formation and brittle fracture, ductile polymers to cutting and chip formation, and elastomers generally exhibit tearing and fatigue. Thus, the erosion behaviour of a wide range of fibre-reinforced polymer composites with different matrix materials has recently been evaluated. These include glass and carbon fibre reinforced epoxy [3–5], polyester [6–9], polyetheretherketone (PEEK) [10], polyetherimine (PEI) [11,12] and polyphenylenesulphide (PPS) [13], to name a few. Typically, the effect of the reinforcement material, concentration, type and orientation as well as its adhesion to the matrix has been studied in these investigations.

Moreover, as many as 20 interrelated test parameter combinations affecting the erosion performance of composites can be established [14]. Typically, the effects of test temperature, particle velocity, impingement angle, erodent material and its size, shape and hardness are studied [14,15]. For example, the effect of the erodent particle size and velocity can be estimated with the aid of

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its kinetic energy  $E_k$ , which has a major role in the erosion behaviour of materials [16]. The kinetic energy of erodent particles can be estimated by Eq. (1) [16]:

$$E_k = \frac{2}{3}\rho\pi R^3V_p^2 \quad (1)$$

where  $\rho$  is the density,  $R$  is the radius and  $V_p$  is the velocity of the solid particles. All in all, the numerous different parameter combinations make it difficult to compare results between different studies.

Stemming from the fact that there are several possible wear modes and mechanisms, many types of wear testing devices exist. Some standard methods are used to rank the wear resistance of different materials, whereas some methods attempt to simulate an actual service environment as closely as possible. The slurry pot test is a commonly used test method to determine the slurry erosion behaviour of different materials on laboratory scale. Slurry pots are simple and easy to operate and can be used in ranking materials based on their erosion resistance as well as to reveal the basic wear mechanisms of the erosion process for a certain material [17,18]. Since the particle flow inside the slurry pot is complicated, it may be challenging to match the particle velocity and impingement angle with a certain, real-life application [18–21]. Therefore, to assess the erosion resistance of materials under hydrometallurgical leaching environments, the testing apparatus design has to resemble the actual application as closely as possible. Computational fluid dynamics (CFD) modelling together with in-situ particle velocity measurements are used to study and validate virtual models of slurry pots [22,23]. CFD model of the test set-up was created and the results were utilised in assessing the flow pattern. However, in this paper, the standpoint is empirical.

This research paper studies the erosion of GF-reinforced VE-FRP using a new in-house constructed erosion test apparatus. The apparatus consists of a pilot-scale reactor where the samples are attached to the agitator blades. The design allows heating of the slurry and the use of acids but in this study only aqueous slurry was employed. The slurry temperature, rotation speed, erodent particle size and concentration varied and the erosion rates of the VE-FRP samples were measured. The worn samples were characterized by scanning electron microscopy in order to evaluate the erosion mechanisms in these rather complicated, two-component composite structures. The results of this study will provide a reference point for the corresponding measurements from tests conducted in sulphuric acid, which will be reported separately.

## 2. Experimental details

### 2.1. Laminates

The tested vinylester composites were manufactured by hand laminating using epoxy vinylester resin (Derakane Momentum 411-350 supplied by Ashland), which is based on a bisphenol-A epoxy resin. It is generally used in applications where good resistance to acids, alkalis, bleaches and solvents are needed [24]. The laminates contained six layers of C-glass chopped strand mat with a nominal weight of 300 g/m<sup>2</sup> each and on both surfaces one layer of 30 g/m<sup>2</sup> C-glass mat. C-glass is typically used in the corrosion barrier layer which is subjected to both aggressive environment and wear. The structure of the laminates is equal to the corrosion-resistant layer generally used in the surfaces of FRP structures applied in acidic hydrometallurgical leaching environments. Because the function of the corrosion-resistant layer is to protect the structural layer from chemical attack, the mechanical properties of the laminate were not considered relevant. The laminates were post-cured for 4 h at 80 °C.

All samples were cut from one laminate and the density, the glass transition temperature and the hardness of the laminate were measured. The density was 1.1416 g/cm<sup>3</sup> and was measured with a Wallace electronic densimeter. The glass transition temperature ( $T_g$ ) of the laminate was 116.8 °C and was measured with a differential scanning calorimeter (DSC), Netzsch DSC 204 model under a nitrogen flow of 50 ml/min at a temperature range of 25–200 °C, according to ISO 11357-1:2009. The Barcol hardness of the laminate was 39 ± 4 and it was measured with a Barber-Colman Impressor GYZJ-934-1 with 45 parallel measurements according to the standard ASTM D2583. The fibre fraction of the laminate was 39.7 wt% and was estimated by means of a residual ash determination test. In this test, a small sample (weight of approximately 1 g) was cut from the laminate and its temperature was raised gradually to 600 °C in an oven. After all the resin had burned away, the remaining ash was weighed and compared to the original weight.

The erosion test samples (size 35 mm x 35 mm) were water jet cut from the laminate and the cut edges of the samples were sealed with vinylester resin (Derakane 441 supplied by Ashland) in order to avoid excess water intake. To cure the Derakane 441 sealing layer and to provide a moisture-independent reference state, the samples were placed in an oven (80 °C) for 6 h and then weighed. To further minimise the edge effects, the cut edges were covered with the sample holder, as demonstrated in Fig. 1b. Two to six parallel samples were tested, depending on the erosion test conditions. After the test, the worn samples were brushed with a soft cloth and cleaned in an ultrasonication bath for one hour to remove any erodent material attached to the sample surfaces and to minimise the amount of loose wear debris that would affect the weight loss determination. Finally, the samples were dried in an oven (6 h at 80 °C), weighed and the weight loss values for each sample were calculated. The wear surfaces were characterized with a scanning electron microscope (SEM; model Zeiss ULTRApplus) using an acceleration voltage of 10 kV. Prior to the SEM studies, the specimens were coated with a thin gold layer to avoid charging.

### 2.2. Test set-up

The erosion wear tests were done with a new in-house constructed erosion test apparatus (Fig. 1a). In this apparatus, the sample holder with the samples was attached to the rotating main shaft that is connected to the motor, located on the top of the lid. A maximum rotation speed of 7.2 m/s at the tip of the blade could be achieved. Four baffles on the inner sides of the reactor walls controlled the flow of the slurry and prevented the sedimentation of the erodent on the reactor walls. The temperature of the slurry could be raised with the heating coils up to 95 °C. No cooling system was built into the system. The slurry temperature during the test was monitored continuously with thermocouples.

The sample holder in this test apparatus resembled one typical agitator type that is used in mixing solids with a liquid. Twelve samples could be tested at the same time and they were attached to the rotating agitator blades (fixed at 45° position), as illustrated in Fig. 1b. One half of the samples were on the pressure side and the second half on the suction side of the blades. It is known that during agitation a different pressure distribution is generated in the pressure and suction sides of the agitator blades. Flow is more uniform on the pressure side of the blade [25,26] although the velocity varies slightly (3.5%) across the sample surface, due to the different local velocity of the blade. On the other hand, on the suction side of the blade, a trailing vortex is created leading to turbulent flow conditions and, consequently, to the localisation of wear [22,23].

In this study, an erodent concentration of 15 wt% in the aqueous slurry was selected since it is typical for many hydrometallurgical processes. However, in order to study the effect of slurry concentration, tests were also conducted using concentrations of 10 wt% and

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