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Delhi iron pillar rust characterization by X-ray diffraction analysis

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Abstract : Rust samples obtained from the region just below the decorative bell capital of the Delhi iron pillar (DIP) have been analyzed by X-ray diffraction (XRD). The identification of iron hydrogen phosphate hydrate in the crystalline form was unambiguous. The stability and formation of crystalline iron hydrogen phosphate hydrate is briefly addressed. Very weak diffraction from the oxy/hydroxides/oxides of iron was found. These phases are most likely present in the amorphous form in the rust. The present XRD analysis of rusts obtained from an inaccessible area of the DIP are compared with earlier analysis of DIP rust obtained from the region accessible to the public.

Key words : *Delhi iron pillar, Rust characterization, X-ray diffraction, Crystalline iron hydrogen phosphate hydrate,Amorphous iron oxyhydroxides.*

INTRODUCTION

The Delhi iron pillar (Fig. 1) is testimony to the high level of skill achieved by the ancient Indian iron smiths in the extraction and processing of iron. It has attracted the attention of archaeologists and corrosion technologists as it has withstood corrosion for nearly 1600 years. Several theories, which have been proposed to explain its superior corrosion resistance, can be broadly be classified into two categories[1-3]: the environmental and material theories. The proponents of the environment theory state that the mild climate of Delhi is responsible for the corrosion resistance of the Delhi iron pillar while, on the other hand, several investigators have stressed the importance of the material of construction as the primary cause for its corrosion resistance. These theories have been critically reviewed elsewhere[1-3]. The role of slag particles in enhancing the passivity in these materials has been earlier addressed[4-7]. In order to obtain insights into the protective passive film that forms on the Delhi iron pillar, relatively old rust samples obtained from the Delhi iron pillar has been characterized by several different techniques[8-9]. The present paper outlines the XRD results.

EXPERIMENTAL PROCEDURE

Rust was collected from several different locations in the region just below the decorative bell capital (Figure 2a) using a plastic scraper. Interestingly, the passive film had healed completely as seen in Figure 2b, which shows the same location nearly a year after the rust samples were taken. This is the region where the rust layer on the exposed surface of the pillar is maximum[8] and therefore, this allowed the collection of a significant amount of rust suitable for characterization by a wide variety of techniques. Some portion of the rust samples thus collected were ground into fine powder and mounted in between two thin polymer foils. A part of the ground powder was also analysed using Fourier transform infrared spectroscopy[10]. The polymer foil containing the DIP rust was used for Mossbauer spectroscopic analysis of the phases present[9]. The same foils were mounted in a