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## Delhi iron pillar rust characterization by fourier transform infrared spectroscopy analysis

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**Abstract :** The oldest rust from the Delhi iron pillar was characterized by Fourier transform infrared spectroscopy (FTIR). The constituents of the scale were  $\gamma$ -,  $\alpha$ -,  $\delta$ -FeOOH, Fe<sub>2</sub>O<sub>3</sub> and phosphate. The FTIR results also revealed that the scale was hydrated. The unambiguous identification of the iron oxides/oxyhydroxides in the FTIR spectrum implies that they are present in the amorphous state as X-ray diffraction (XRD) analysis did not reveal these phases. The results of the present study are compared with earlier FTIR spectroscopic results of atmospheric rust formation.

**Key words :** Delhi iron pillar, Rust characterization, Fourier transform infrared spectroscopy, Phosphates, Amorphous iron oxyhydroxides.

### INTRODUCTION

The precise reason for the corrosion resistance of the famous 1600 year old Delhi iron pillar (Fig. 1)<sup>1</sup> is not well understood<sup>2,3</sup>. In order to gain insights into the nature of the passive film that forms on the Delhi iron pillar (that is responsible for its excellent resistance to atmospheric corrosion<sup>4,5</sup>), detailed characterization studies of the undisturbed rust from the region just below the decorative bell capital of the Delhi iron pillar (DIP) were undertaken. The X-ray diffraction (XRD) pattern of the rust revealed the presence of crystalline iron hydrogen phosphate hydrate (FeH<sub>2</sub>P<sub>2</sub>O<sub>7</sub>·4H<sub>2</sub>O which can also be viewed as FePO<sub>4</sub>·H<sub>2</sub>PO<sub>4</sub>·4H<sub>2</sub>O)<sup>6</sup>. The XRD analysis also indicated the presence of a very small amount of iron oxides/oxyhydroxides in the crystalline form<sup>6</sup>. However, as the brown rust samples did not possess the characteristic reddish-yellow colour of iron phosphates, it was apparent from visual observation that the rust contained a significant amount of iron oxides/oxyhydroxides in addition to the iron hydrogen phosphate hydrate. Moreover, earlier XRD analysis<sup>6,7</sup> of rust samples taken from the lower regions of the pillar, which was in contact with humans, report the presence of iron hydroxides/oxides in the rust samples collected from the top regions of the pillar was anticipated. It was also evident that in case the iron oxides/oxyhydroxides were amorphous in nature, they would not provide any diffraction peaks and therefore XRD is not the characterization technique that will reveal the presence of these amorphous phases in the rust.

As it was clear that a significant portion of iron oxides/oxyhydroxides were not present in crystalline form in the DIP rust (based on the XRD pattern obtained from the Delhi iron pillar rust<sup>6</sup>), it was necessary to employ other characterization techniques that will be capable of identifying iron oxides/oxyhydroxides even if they are present in the amorphous form. The characterization technique which will reveal the different allotropic modifications of iron oxides and oxyhydroxides even if they are amorphous in nature is Fourier