



# Electrochemical and anticorrosion behavior of functionalized graphite nanoplatelets epoxy coating



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## ABSTRACT

Functionalized graphite nanoplatelets (FGNP) were used as efficient and compatible nano-particles to produce homogenous epoxy nano-coating with impressive anticorrosion behavior for carbon steel. The characterizations of the nano-particle and nano-coating were carried out by SEM, FT-IR, XRD, TEM and pull-off test. Fine distribution of nano-particles in the cured nano-coating with particle sizes of 20–40 nm was obtained. Electrochemical experiments, salt spray and X-ray fluorescence showed that the nano-coatings protect the metal substrate by formation of passive layer and physical barrier characteristics. Three samples (0.25%, 0.5% and 1%) of FGNP-epoxy coatings were prepared that 0.5% showed better anticorrosion properties.

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## 1. Introduction

Different corrosion protection methods have been used for metal substrates. Organic coatings are the most common and cost effective methods and among these, organic nano-coatings show substantial property enhancement at much lower loads than polymer composites with conventional micro-scale fillers [1].

In recent years, graphene and derivatives such as graphite nano-platelets (GNPs) have garnered the most interest for nano-materials [2]. Unique mechanical, electrical, thermal conductivity, thermal stability, chemical inertness, electromagnetic interference (EMI), gas barrier properties and impermeability to small ions and molecules, make them ideal candidates for different applications [1–5].

GNPs are not individual graphene sheets but comprise multiple stacks graphene layers. While graphene and graphene oxide (GO) contain a monolayer of carbon atoms arranged in a two-dimensional lattice, GNPs have several graphene layers, a high aspect ratio and are prepared using cost-effective method [4].

Some studies on graphene and GO have examined corrosion protection of metal substrates. In earlier studies, chemical vapor deposition (CVD) technique was an interesting method to grow graphene on metal surfaces [6–8]. For the first time, Chen [9] used graphene as a passive layer for protection of Cu and Cu/Ni alloys and found that the metal surface was well-protected from oxidation even after heating up to 200 °C in air for 4 h. Electrochemical studies in aqueous media for graphene coating of Ni and Cu, displayed a sharp decrease in anodic and cathodic reaction rates [6].

Prasai [10] demonstrated that single-layer and multilayer graphene film could serve as a corrosion inhibiting coatings for Cu and Ni metals, with 7 times and 20 times less corrosion rate for graphene/Cu and graphene/Ni, respectively compared to bare metal.

All of these researchers considered CVD to be a large area direct deposition of ultrathin graphene film on metal surface that suffers from partial oxidation at graphene grain boundaries, cracks or defects in the graphene film, high temperature (650–1000 °C) process, high vacuum requirement and limitation on sample size [11].

It was necessary to develop protocols to grow highly uniform defect-free large-grain graphene films [7,10] to overcome these problems. Researchers developed electrodeposition and electrophoretic techniques [12]. The electrophoretic deposition technique was first used by Wen [13] to reduce GO deposition on the metal substrate, which proved to be an effective inhibitor of anodic

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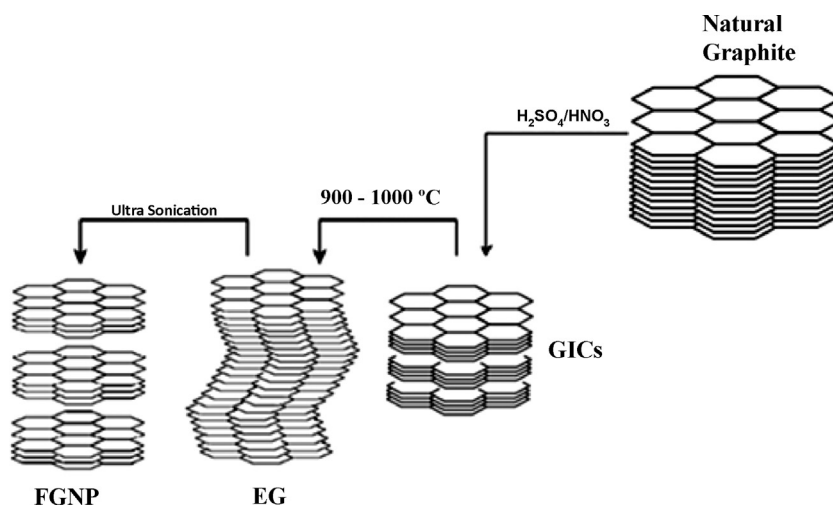


Fig. 1. Schematic process of preparing FGNP from GICs.

reaction. Singh [14] and Sahu [15] developed electrochemical approaches for deposition of graphene nano-sheets on Cu in chloride environments. Singh decreased the corrosion rate by a factor of 10 and Sahu by a factor of 18 over bare Cu. Singh et al. [11] prepared a hydrophobic GO polymer coating treated with silicon fluid on Cu in a chloride environment. Electrochemical degradation of this system was 3 orders of magnitude lower than bare Cu.

Natural graphite flakes are difficult to exfoliate into GNPs unless they are intercalated by appropriate molecules or ions, such as sulfuric or nitric acid, metal chlorides and alkali metals. This intercalated product is known as a Graphite Intercalated Compound (GIC) [1]. Acid treatment of graphite, increases its interlayer spacing, weakening the interlayer interactions and facilitating the exfoliation of the GIC by thermal methods and also oxidize the GIC to a far less degree than GO [1]. So GIC is heavily oxygenated (bearing hydroxyl, epoxide, diol, ketone and carboxyl functional groups) [1,2]. GNPs can initially be prepared using GIC [2]. GIC exfoliates by rapid heating to form a large expansion (up to hundreds of times) worm-like accordions with low density that is called exfoliated or expanded graphite (EG) [1].

Thermal expansion of GIC in an inert gas (argon) can reduce larger amounts of oxygenated groups, but does not remove all of them and retains some specific amounts of oxygenated groups on EG that after sonication of EG in a proper solvent the produced GNPs (30–80 nm), is called Functionalized graphite nanoplatelets (FGNP) [1,16]. FGNP is a mild oxygenated Graphite NanoPlatelets, and this feature of FGNP was properly used in this study. Because Pristine graphene and graphite are not compatible with polar organic polymers and do not form homogeneous composites except in the presence of a dispersion agent (because of having no oxygenated groups) and on the other hand, the presence of too many oxygen groups make the GO sheets, strongly hydrophilic and electrically insulating, but FGNP is an intermediate product. The presence of some oxygen functionalities in FGNP, allows it to be used as promising carbon nano-filler with ideal dispersion in polar polymers such as epoxy. On the other hand FGNP is electrically conductive and less hydrophilic [1,2].

In this work, FGNP was used as a proper functionalized nano-particle with good dispersion and anticorrosion behavior in epoxy binder on carbon steel.

Thus far, no long term and detailed investigations of FGNP-epoxy coating anticorrosion properties and mechanisms on a carbon steel substrate have been done.

## 2. Experimental

### 2.1. Materials

The purchased graphite (GICs) is an expandable sulfuric acid-intercalated graphite Spec: 9950250 (Boading Action Carbon Co., Ltd), epoxy resin EPIRAN6 (EEW 185-196) was purchased from Khuzestan petrochemical Co, H46 amine hardener (H active:100) from Pars Gohar Co, epoxy thinner T51 was purchased from Rangin Zereh Co. Carbon steel (ST37) plates were used as base metal for the coatings. Sodium chloride purchased from Merck Company, Aqueous solutions were prepared from double distilled water.

### 2.2. Preparation of functionalized graphite nanoplatelets

FGNP were prepared by the rapid thermal expansion (for one minute) of GICs at 900 °C under inert argon atmosphere to form EG. Sonication of EG was carried out according to [16], within (water/ethanol (25/75 (%v/v)) solution in an ice bath for 8 h followed by drying in an oven at 50 °C for 48 h to produce FGNP. The process of preparing FGNP from GICs is shown schematically in Fig. 1.

### 2.3. Preparation of functionalized graphite nanoplatelets epoxy coating

FGNP powder was first dispersed in epoxy thinner (T51) by sonication for 1 h, subsequently added into the epoxy resin and mixed for 2 h to ensure a homogeneous dispersion. Finally, amine hardener H46 curing agent was added to the mixture. The FGNP epoxy coatings were applied by air spraying over sand blasted (ASTM D4417) carbon steel panels of 15 cm × 10 cm and were evaluated after 14 days of curing at room temperature. The thicknesses of the dry films after subtracting sand blast profile (20 μm) were obtained 75 μm.

### 2.4. Evaluation of nano-particles and nano-coatings

FGNP and EG morphologies were investigated using a AIS-2100 SERON Co scanning electron microscope (SEM), for investigation of surface functionalized groups on (GIC, EG and FGNP), FT-IR spectra were taken with KBr pellets using a Bruker alpha FT-IR spectrometer in the range of 4000–500 cm<sup>-1</sup> and for identification of FGNP dispersion state in epoxy resin and its crystal structure, X-ray diffractions of FGNP and FGNP epoxy coatings were analyzed

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