NiO synthesis NiO-based oxide powders were synthesized by different methods in order to evaluate the sensitive performance for different test gases and also to correlate the sensitive properties with the structural, morphological and surface chemistry properties.

1. NiO1 - Method 1 - NiO was obtained by a hydrothermal synthesis method from hydrated Ni nitrate as Ni (NO3) 2, as an inorganic precursor for Ni, water as solvent, ethylene glycol (EG) as structural targeting and ethylenediaminetetraacetic acid (EDTA) as a complexing agent. The mixture obtained by dissolving the above-mentioned reagents in water was kept under continuous stirring for 24 hours at room temperature, followed by hydrothermal treatment at 180 ° C for 24 hours. The resulting precipitate was filtered and washed with water and ethanol, dried at 100 ° C, and heat treated in air at 400 ° C for 8 hours. Subsequently, some of the powder was heat treated at 500 ° C for 8 hours in air. The purpose was to track changes in crystal structure, morphology, and surface chemistry. Given that the OHads groups on the surface (detected by XPS) have an important role in the sensing mechanism, practically the degree of hydroxylation of the surface, we performed this heat treatment at a higher temperature to monitor their weight. The samples were graded NiO-1\_400 and NiO-1\_500, respectively.

2. NiO2 - Method 2 - This method of preparation also addresses a synthesis protocol involving hydrothermal treatment. In this case we used Ni acetylacetonate (Ni (C5H7O2) 2), ethanol solvent, urea (CO (NH2) 2 as hydrolysis agent, Tripropylamine (TPA) as structural targeting agent, Polyvinyl pyrrole (PVP) as a precursor for Ni.) as a stabilizer and tetrabutyl ammonium hydroxide (TBAOH) as a pH mediator NiAcAc was dissolved in ethanol, then PVP was added. at ~ 11 with TBAOH The solution obtained was left under continuous stirring until the next day for maturation, then hydrothermally treated at 180 ° C for 24 hours The hydrothermally treated solution was left at ambient temperature for 3 weeks, until A gel with a resin-like gel was dried at 80 ° C under vacuum for 2 hours and then heat-treated at 400 ° C for 8 hours in air, as the amount obtained was relatively small (0.44 g). heat treatment was performed at a temperature of 500 ° C as in the case method 1. The sample was marked with NiO-2\_400.

3. NiO3 - Method 3 - In this case we developed through a self-assembly method, obtaining a NiO modified with Graphene nanoplatelets (GNP). As a precursor of Ni we used nickel acetylacetone (NiAcAc) and as solvent water and ethanol in a ratio of 1: 1. NiAcAc was dispersed in a mixture of water and ethanol, with vigorous stirring (1000 rpm), at 40 ° C for 30 minutes, after which Triton X100 and Tripopilamine were added as structural targeting agents, hydroxide. tetrabutyl ammonium (TBAOH) for complete solubilization of Ni and GNP ions. The obtained mixture is kept at room temperature with stirring for 48 hours, ultrasonically 30 minutes for very good dispersion of graphene and to avoid the formation of Ni agglomerates. After drying, the black powder is subjected to heat treatment at 400 ° C and 500 ° C, respectively, for 6 hours in air. The samples were noted: NiO\_rGO\_400 and NiO\_rGO\_500 respectively