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# TERNARY NANOCOMPOSITES BASED ON OXYSULFONATED ONION-TYPE NANOCARBON MATERIALS/POLYVINYLPYRROLIDONE AND CARBON BLACK FOR SURFACE ACOUSTIC WAVE HUMIDITY SENSORS

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## *Introduction*

Carbon nano-onions (CNOs) were first synthesized by Ugarte in 1992 through electron irradiation of carbon black. Typically, they are produced by annealing diamond nanoparticles at high temperatures in an inert atmosphere. CNOs are zero-dimensional nanoparticles (1.4–50 nm) with closed multilayer shells and a fullerene core. Traditional methods yield hydrophobic CNOs, but oxidation with nitric acid or ozone introduces polar groups (carboxyl, hydroxyl, carbonyl), enhancing their solubility in polar solvents like water and methanol. These functionalized CNOs retain their structure and enable further reactions. Applications include electrochemistry (supercapacitors, batteries), catalysis, medicine, and gas sensing. This work focuses on creating sensitive layers for surface acoustic wave (SAW) sensors, which consist of a piezoelectric substrate, interdigital transducers, and a gas-sensitive film for monitoring relative humidity.

## *Materials and methods*

The process for obtaining oxysulfonated onion-type nanocarbon materials (ox-CNOs-$SO_3H$) is as follows:

- *Synthesis of CNOs* - nanodiamonds are heat-treated at 1650°C in He atmosphere.
- *Oxidation of CNOs* - CNOs are treated in Ar-$O_2$ plasma (2:1 volumetric mixture) at 10 torr and room temperature, with a 5-min. injection and 5–10 min. of exposure.
- *Preparation of ox-CNOs-$SO_3H$* - 50 mg of ox-CNOs was dispersed in 10 mL of 95% $H_2SO_4$ in Teflon autoclave and heated at 200°C for 20 hours. The product was washed with deionized water until neutral and vacuum-heated at 150°C for 6 hours.

*Preparation of the sensitive layer involves the steps:*
- the quartz substrate was cleaned in an ultrasonic bath with ethanol and demineralized water for 10 minutes,
- 3 mg of polyvinylpyrrolidone is dissolved in 15 mL of deionized water under magnetic stirring for 20 minutes,
- 6 mg of ox-CNOs-$SO_3H$ was added to the solution and stirred for 90 min., 1 mL of 10% aq. carbon black dispersion was added, and stirring continued for 180 min.
- the dispersion was deposited on the substrate via spin coating (3000 rpm, 40 s).
- the film was heated at 100°C for 60 minutes, followed by a final heat treatment at 200°C for 30 minutes.
- the sensitive layer was dried in a vacuum oven at 50°C for 24 hours.

***Results and conclusions***
The sensor utilized is of the "dual delay line" type and constructed on a piezoelectric quartz substrate. It features a double delay line to compensate for thermal drift. Specifically, one delay line is coated with a ternary nanocomposite sensitive to relative humidity (RH) variations, while the second delay line consists of the piezoelectric substrate without a sensitive layer. The sensitive films are deposited onto the piezoelectric quartz substrate using the "spin coating" method (Figure 1).

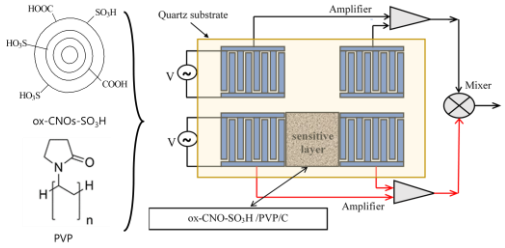


**Figure 1**. Design of the SAW humidity sensor

The use of this ternary nanocomposite offers several significant advantages. The presence of ox-CNOs-$SO_3H$ provides a high specific surface area-to-volume ratio, strong affinity for water molecules ("mass loading"), and a change in the resistance of the sensitive layer upon interaction with water molecules ("electric loading").
The sensor has good mechanical properties and reliable detection across a wide temperature range. The hydrophilic nature of PVP and ox-CNOs-$SO_3H$ enhances interaction with water molecules. Carbon black improves the dispersion of ox-CNOs-$SO_3H$ within the polymer matrix and modulates the conductivity of the moisture-sensitive layer, acting as an effective filler.

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