



Short communication

## Self-assembled micro-structured sensors for food safety in paper based food packaging



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### ABSTRACT

Natural self-assembled microstructured particles (diatomaceous earth) were used to develop a gas sensor paper with detection mechanism based on visible and distinct color changes of the sensor paper when exposed to volatile basic nitrogen compounds. The coating formulation for paper was prepared by applying diatomites, polyvinyl alcohol (PVOH), and pH sensitive dyes on acidic paper substrate. The surface coating was designed to allow a maximum gas flow through the diatomite sensors. The produced sensor paper was tested for sensitivity using different ammonia concentrations and we observed a sensitivity lower limit at 63 ppm. As a comparison, the results show comparable sensitivity levels to carbon nanotube based sensor technologies reported in literature.

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### 1. Introduction

Food safety is one of the major health issues facing the global economy and is gaining an increasing amount of awareness in the biosensor and food packaging area. It is reported that each year a total of 48 million Americans become sick from contaminated food. Also, an estimated 128,000 of these cases do require hospitalization and 3000 cases result in death [1]. The economic effect of foodborne illnesses and food safety in the US is an estimated burden of \$77.7 billion each year [2].

An efficient detection of microbial contamination, chemicals and toxins in food is the solution to the prevention and recognition of problems related to health and safety [3]. Although, science and technology has recently advanced very sophisticated technologies to detect foodborne threats, including the latest development in nanotechnology, scalable low cost and rapid detection methodologies are still lacking [4,5].

The traditional techniques such as culture and colony counting methods, immunology-based and polymerase chain reaction (PCR) based methods unfortunately take up to several hours or even a few days in order to reach a result [6–8]. Spectrometric methods have also been developed [9]. These methods are rapid, however, require expensive equipment and trained personnel for evaluation [10–12]. Clearly this is inadequate for certain applications, and recently many

researchers are focusing towards the progress of rapid, less costly and more qualitative first response methods [13–15].

Novel bio-molecular techniques for food pathogen detection exist and are currently being developed to improve the characteristics of food biosensors. Key attributes include sensitivity, selectivity, and detection speed [16–20]. The sensor solution has to also be robust and reliable, effective and suitable for in-situ analysis [21–23]. New technologies show high potential but further research and development is needed in order to have low cost, accurate and rapid detection methods that can be integrated to the food supply chain and thus to improve the overall food safety of the global system [24–28].

Food packaging is an essential medium for preserving food quality, minimizing food wastage and reducing preservatives used in food. The packaging also provides the containment protection against physical or chemical damage and gives critical information to the end users the consumers and distributors on the content and its use. Integration of sensor technology to food packaging will provide the food safety related element that can dramatically increase the total safety of the food supply chain.

The objective of this research was to study the use of diatomaceous earth as a micro-structured media for food safety sensors. The naturally occurring diatomaceous earth consists of fossilized remains of diatoms that are types of algae with a hard-shell structure. Traditionally diatoms have been used in paper based materials, as for example filler in paper making, filtration media, mild abrasive, absorbent material, and a thermal insulator. Perhaps its most famous application is for the stabilization material for dynamite [29]. The functionality of gas sensor paper is based on pH sensitive dye that indicates spoilage of meat or fish

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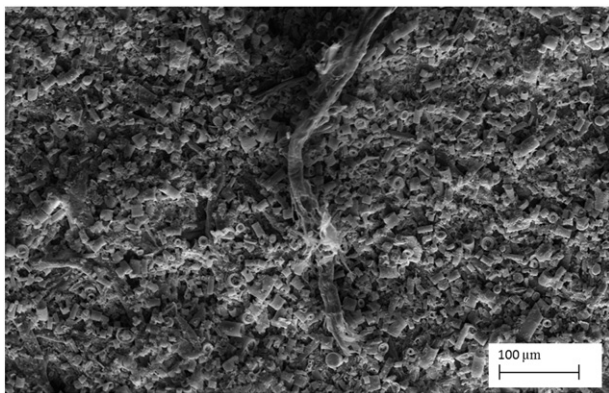


Fig. 1. SEM image of sensor paper surface structure with 500× magnification and coat weight of 6 g/cm<sup>2</sup>. A cellulose fiber can be seen in the middle of the image.

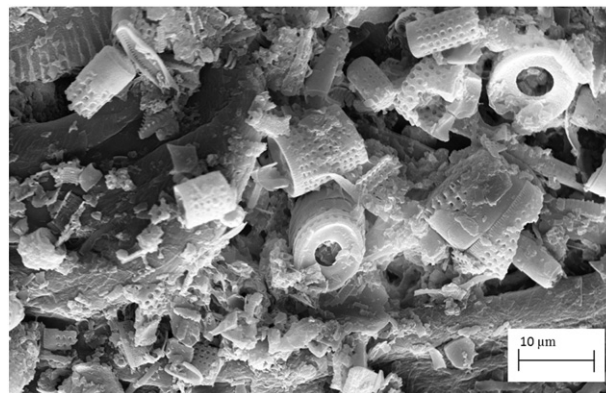


Fig. 3. SEM image of the sensor paper surface using SEM and with 4000× magnification and coat weight of 6 g/cm<sup>2</sup>. The barrel like structure and micron sized open pores are clearly visible.

products by color change. The reaction depends on the chromogenic material. For the detection of ammonia, produced during spoilage, pH indicators such as bromophenol blue or bromocresol purple can be used [30,31]. The ammonia gas acts as a Lewis-base and induces the color change due to hydrogen release. Other gasochromic materials are complexes and their color change is induced through changes in the ligand field [32]. By adding the diatomaceous earth material as a medium to the sensor paper, we developed a low cost, highly selective and sensitive detection method that can easily be integrated in the paper production process.

## 2. Experimental section

### 2.1. Materials

Diatomite, supplied by World Minerals (Diafil295), had an average surface area of 30.1 m<sup>2</sup>/g. The diatoms used for this research composed of 86–93% SiO<sub>2</sub>. Polyvinyl alcohol (PVOH) was supplied by Sekisui Specialty Chemicals (Selvol 523) and bromocresol purple, a pH sensitive dye from Alfa Aesar, was used to create the coating for the sensor paper. Acidic paper was used as a substrate for the sensor paper prototype development.

### 2.2. Coating formulation

PVOH was selected as a binder in the coating formulation because of its high binding power and high water absorption capacity. The solution of PVOH was prepared at 20% solids by adding the required amount of



Fig. 2. SEM image of sensor paper surface structure with 1000× magnification and coat weight of 6 g/cm<sup>2</sup>.

dry PVOH powder to cold water under agitation and heating the mixture to 85 °C. A de-foamer was added before the PVOH addition. The solution was held at this temperature for 35–40 min to assure complete dissolution and hydration of PVOH. The PVOH solution was cooled to 25 °C before addition to the slurred diatomite particles at a pigment to binder ratio of 5:1 (pigment: 100 parts and binder: 20 parts) under slow agitation. Finally, pH sensitive dye is added into the coating formulation between 0.05 parts and 1 part. The coatings were mixed in a laboratory mixer for 20–30 min under low shear to ensure that no air is entrapped into the coating and cause foaming. Application of the coatings on acidic paper was made with standard industry technique of drawdown rods that give users the ability to fine-tune coating thickness quickly and easily. The coat weights were varied by rod number and the coating solids content. The coat weights applied were between 6 and 20 g/m<sup>2</sup>.

### 2.3. Surface characterization and sensor testing

The prepared samples were analysed using optical microscopy. Scanning electron microscopy (SEM, Zeiss DSM 940) equipped with an energy dispersive spectroscopy (EDS) detector was used to observe the surface morphologies of the coating, its thickness and diatom structures. A profilometer (Bruker/Veeco Dektak 150) was also used

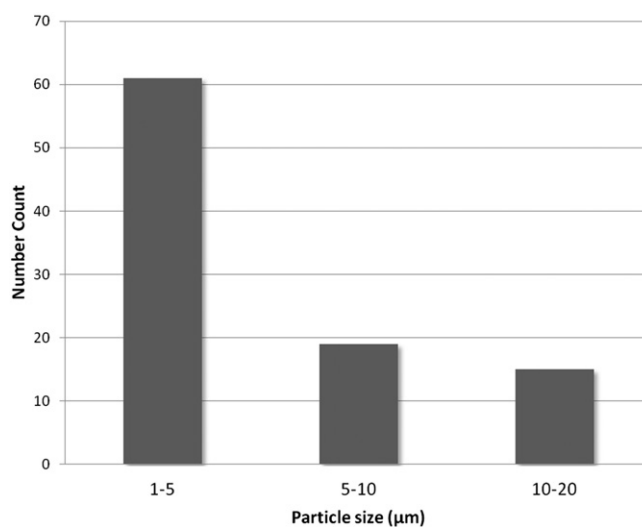


Fig. 4. Averaged particle surface size distribution of diatomite coated paper with coat weights of 6 g/cm<sup>2</sup>.

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