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**PAPER** Mehmet E. Solmaz *et al.* Smartphone-based detection of dyes in water for environmental sustainability

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## Smartphone-based detection of dyes in water for environmental sustainability†

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We report the applicability of an ultra-low cost, field-deployable, plastic fiber based smartphone spectrometer system to study dye adsorption from water. A custom cradle using the smartphone's built-in flash and camera was designed for visible absorbance spectroscopy without any need for external electrical components. We firstly investigated the performance of the smartphone spectrometer for methylene blue (MB) absorbance in water with a detection limit of 0.5 ppm, and compared it to that of a commercial spectrometer. We elaborated on the contribution of image formats to the calibration of the standard absorbance curve. We then studied the catalytic activity of electrospun polyacrylonitrile (PAN)/zeolite composite nanofibers by analyzing MB adsorption as a function of time. The results obtained in the proposed compact, cost-effective and high-performance platform can help transform measurement science for sustainable water management.

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## 1 Introduction

Possible accumulation of dyes in a body of water is harmful to humans and the environment. Their removal at industrial facilities is extremely important before they are discharged to the environment. Adsorption is one of the most effective ways to remove undesirable molecules from wastewater,1,2 and absorbance spectroscopy is the major measurement method to observe dye removal efficiency.3,4 Cost effective field-portable measurement devices provide an alternative way for controlling the removal of dyes for environmental sustainability and management. In particular, smartphone platforms with integrated sensing systems are drawing huge interest due to the premises of replacing their benchtop counterparts with compatible performance and the ability to use them in a resource-limited setting. Ultimately, smartphone instrumentation has gone beyond microscopy to replace commonly used instruments such as colorimeters.5 For instance, a smartphone by itself is enough to quantify colorimetric test strips without any external attachments,6,7 or with a simple holding attachment for a water monitoring system.8 By adding external 3D printed housing and optical components, a colorimetric plate reader based on image processing was proposed for enzyme-linked immunosorbent assays (ELISA).9 On the other hand, smartphone based spectrometric and colorimetric analyses were studied using diffraction gratings as dispersive elements. Here the color spectrum image is further processed in various color spaces to extract quantitative data. The wavelength spectrum of interest is usually limited to 400-700 nm due to optical filters placed in front of the camera by the manufacturers. A smartphone ELISA platform capable of biomarker absorption analysis was demonstrated using a custom cradle consisting of several optical components and an external broadband source.10 Similarly, a smartphone fluorimeter system was designed to detect miRNA sequences in a liquid based assay using an external laser as the source.11 Optical fibers were incorporated onto smartphone spectrometer systems due to their precise light confinement and flexible nature. A fiber-based endoscopic spectrometer integrated onto the camera sensor was proposed for food quality monitoring.12 Recently, smartphone spectrometers with no external electrical and optical components were proposed to improve portability, lower the costs, and help environmental sustainability. A surface plasmon resonance based refractive index sensor by assembling a fiber optical bench on top of a smartphone with no external LEDs was reported.13 A spectrometer relying only on a smartphone's built-in flash as the light source and a compact disk as the reflection grating was used to detect glucose and troponin I.14 No prior smartphone spectrometer/colorimeter instrument has been fully integrated with plastic optical fibers, collecting the light from the built-in flash and delivering the transmitted light to the camera sensor on the phone as the detection instrument to study the adsorption efficiency of electrospun nanofibers for the removal of dye from waters.



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#### **Analytical Methods**

Here, we report an ultra-low cost, portable, plastic fiber based spectrometric and colorimetric smartphone platform to study dye adsorption by electrospun polyacrylonitrile (PAN)/zeolite nanofibers for field-deployable environmental and wastewater management. The composite nanofibers provide an advantageous combination of zeolite particles and electrospun PAN nanofibers. Electrospinning is a versatile and efficient fabrication technique for nanostructured fibers of both organic and/or inorganic materials.<sup>15,16</sup> The resulting nanofibers enable not only a high surface area to volume ratio, but also superior mechanical integrity compared to the powdered materials.<sup>17,18</sup> These properties make PAN/zeolite composite nanofibers optimal candidates for adsorption applications. Adsorption of methylene blue (MB) molecules on large-area nanofibers decreases the absorbance of the liquid that is picked up by the camera sensor. To enable absorbance measurements, we have designed a custom cradle that aligns plastic optical fibers onto phone optical components. While the cradle is fixed, the liquid can be inserted into the holder slot. The proposed platform is straightforward and achieves high resolution by using all plastic components without the need for any external emissive sources, electrical sources and cables, and lenses. Our work enables smartphone based adsorption studies of other molecules in a robust and economically advantageous setting.

## 2 Experimental methods

#### 2.1 Smartphone spectrometer system

A photographic image of the smartphone based spectrometer is shown in Fig. 1a. The smartphone spectrometer was manufactured to integrate with LG G4 (1/2.6" sensor size with 5312  $\times$  2988 resolution, 1.12  $\mu m$  pixel size) by using a rear-facing camera as the digital light detector. A custom-designed cradle, consisting of a hot-plug apparatus toting a diffraction grating, and a smartphone case holder keeping the whole part compact with the cuvette holder were fabricated from the Acrylonitrile Butadiene Styrene (ABS) polymer using a 3D printer (Zortrax M200). The total polymer usage was about 150 g.

The system carries two pieces of plastic (PMMA) fibers: a 1.5 mm diameter fiber couples the light from the cellphone flash to the cuvette and a 0.25 mm diameter fiber collects the transmitted light, which passes through a linear diffraction grating (1000 lines per mm, Rainbow Symphony) at an angle of  ${\sim}42^{\circ}$  to the normal to reach the smartphone camera. The diffracted light is the first-order spectrum and the zeroth order is steered away from the camera on purpose. The designed spectrometer system is simplified with no collimating lens or mirror components in the light pathway and all components are mechanically aligned with the smartphone flash and camera, and with each other. The total cost of the components is less than \$10 with the biggest percentage being for the 3D printer material. The main advantage of using plastic optical fibers instead of glass-based optical fibers besides the cost is the ability to use them without any special tools for stripping and cutting. Although the plastic fibers do not have a core with a different refractive index, the light is guided due to the air cladding.



Fig. 1 (a) A photograph of the smartphone spectrometer with the inset (top-left) of a plastic fiber coupled to the built-in flash. For more information on the design and assembly of the system, see Fig. S1, ESI.† (b) The spectral images obtained by using different-sized plastic fibers. (c) The output spectrum comparing the size of fibers used in the smartphone with that of a commercial spectrometer. (HSV) means the value is plotted.

#### 2.2 Digital image processing

A digital camera uses a two-dimensional array of pixels to record an image. When light falls on the pixel, the intensity value is calculated with the number of photons stored in the pixel. As the pixels are not capable of sorting photons in terms of color, only grayscale images could be created. To capture a color image, the color filter array is placed over each pixel that permits only red (R), green (G) or blue (B) primary colors. When light passes through the color filter, only filtered photons are stored in the pixel which gives the intensity value for the respective color. Therefore, each pixel carries single color information: R, G or B. This means one color value at any pixel location is known creating a RAW image. Other missing two color values can be calculated by interpolating from nearby neighbours where those colors are known. By this process, named as demosaicing, RGB values at each pixel location are calculated. After applying post-processing methods, such as white-balance, gamma correction, color space correction and compression, images are converted to a commonly used format like JPEG.

Although JPEG image formats have advantages like occupying a small size and being instantly displayable, there are certain drawbacks like information loss due to the compression,

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Fig. 2 (a) The photograph of MB solutions from 0.1 to 10 ppm; (b) absorbance spectra obtained from smartphone and commercial spectrometers for serial dilutions; (c) the absorbance at 664 nm was measured by using the smartphone spectrometer in RAW and JPEG formats and one commercial spectrometer. LOD was calculated as three standard deviations above the distilled water measurement.

having a non-linear RGB color space,<sup>19,20</sup> and providing only an 8 bit color depth. On the contrary, RAW images store original image data without any post-processing and provide 10–14 bits color information. The post-processing steps, which are applied to the RAW image, corrupt the linearity of the information. In a linear image, the intensity value of each pixel is directly related to the number of photons received at that location and is essential for quantitative scientific data acquisition as a linear relationship with scene radiance is maintained.

The RAW format is a linear image, but it is not straightforward to work. Therefore, many models are proposed to approximate raw (linear) RGB from nonlinear RGB images based on a series of training images taken under different settings and light conditions as well as ground-truth raw images.<sup>21</sup> Although it is reported that this non-linearity can be overcome by simple transformations for gamma correction,<sup>22</sup> it has not been verified by other researchers.

In this study, the RAW image format was used to overcome the aforementioned limitations of the JPEG format as using raw data enables precise image analysis. The images were processed both in RAW and JPEG formats and the impact of formats on image analysis was demonstrated. The key question is how to get a RAW image using a smartphone. Most semi-professional and professional cameras have the capability to store the images in a RAW format, which is unconventional in smartphones. However, recent developments in smartphones provide access to raw image data free from postprocessing.<sup>23</sup>

There are a variety of RAW file formats such as ".NEF" (Nikon), ".CR2" (Canon) and ".DNG". Here the ".DNG" format was studied as it is a common open format. Since there is no smartphone app for processing ".DNG" images in a smartphone yet, the images were transferred to a computer to process in the Matlab environment. To test the reliability of the camera, five consecutive images were captured in JPEG format. The difference in the normalized values of images is found to be  $\pm 2.73\%$  with respect to the average. Therefore, the average of five consecutive images was found to be adequate and was used in processing and analyzing the experiments in order to improve the reliability of the results. Note that all the images were captured under the same conditions such as the same shutter speed and ISO, and the manual mode is selected to prevent any possible post-processing performed by the smartphone.

After images are captured in ".DNG" format, they were first converted to tagged image file format (TIFF) for easier extraction of the R, G, B values of the image with freely available DCRAW software,<sup>24</sup> which preserves the linear relationship between RAW images and the radiance scene. Then, these RGB values were transformed to HSV (Hue–Saturation–Value) as HSV is more robust to illumination variation. Among the H, S and V, the V (value) was employed for processing the image. The value means, for this study, the amount of the transmitted light through the solution. After the line (column or row which corresponds to the maximum irradiance) is selected in the image, the value *versus* wavelength is plotted.

For the conversion of each pixel index in the image into its corresponding wavelength value, a calibrated fiber-optic



Fig. 3 SEM micrographs of (a) zeolite powder, (b) PAN nanofibers, (c) PAN/zeolite nanofibers, and (d) FTIR spectra of the materials.



**Fig. 4** (a) The effect of different fiber catalysts on the catalytic degradation of MB (Inset: the color bands observed on the smartphone screen at initial and 15 minutes). (b) The photographic image showing the color of the fibrous mats and the resultant solutions after adsorption.

spectrometer (HR2000; Ocean Optics, FL, USA) was used with a fluorescent lamp. We used a similar calibration method described in ref. 25, assumed that there is a linear relationship between the wavelength value and pixel index, and extracted spectral resolution values for different size plastic fibers. For the same cradle geometry, we obtained different spectral images of the same fluorescent lamp that show different spatial resolutions (Fig. 1b). A 0.25 mm plastic fiber provides a resolution of 0.26 nm per pixel while using larger diameter 0.5 and 1.0 mm fibers gives spectral resolutions of 0.265 nm per pixel and 0.27 nm per pixel, respectively. Although the spectral line shapes obtained from different sized fibers show fluorescent light characteristics with 4 peaks between 400 and 700 nm, the spectrum with the 0.25 mm plastic fiber closely resembles the spectrum of the calibrated spectrometer (Fig. 1c). The spectral resolution and signal-to-noise ratio are directly related to the fiber diameter.

## 2.3 Fabrication of PAN/zeolite composite nanofibrous membranes

Polyacrylonitrile (PAN,  $M_w = 150\ 000\ \text{g mol}^{-1}$ , Aldrich) solutions were prepared by dissolving 15 wt% PAN powders in *N*,*N*-dimethyl formamide (DMF, Aldrich) at room temperature for 1 day. PAN/zeolite solutions were prepared by adding zeolite powder into the PAN solution. The composite mixture was stirred for 1 h at room temperature. The concentration of zeolite in the polymer solution was 10 wt%.

The PAN and PAN/zeolite nanofibrous membranes were prepared using a commercial platform (Inovenso Basic Setup). The viscous dispersion was loaded in a plastic syringe connected with polyethylene tubing. The feeding rate of the polymer solution was kept at  $1.5 \text{ mL h}^{-1}$  for PAN and  $3.0 \text{ mL h}^{-1}$  for PAN/zeolite nanofibers. A potential of 14 kV was applied at a tip-to-collector distance of 20 cm, and the electrospinning time was fixed to 7 minutes to ensure equal thickness. The relative humidity and temperature were 55% and 26 °C, respectively.

The surface morphology and diameter of the nanofibers were observed by using a scanning electron microscope (SEM, FEI Quanta 250 FEG). For quantification of average fiber diameters, Fiji-Image J software was used. The elemental composition of

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the prepared materials was also elucidated using Energy Dispersive X-ray (EDX) analysis. Attenuated total reflection (ATR) infrared (IR) measurements of zeolite, PAN and PAN/ zeolite nanofibers were performed with a Thermo Scientific Nicolet iS5 FTIR spectrometer. The contact angle (CA) measurements were carried out on the nanofibers with a KSV Attension Theta Lite Optical Tensiometer.

#### 2.4 Adsorption studies of PAN/zeolite composite nanofibers

The catalytic activity of the zeolite in the form of a nanofibrous mat was examined employing a model redox reaction of methylene blue (MB, Merck) and sodium borohyride (NaBH<sub>4</sub>, Aldrich). For the reduction of MB, a mixture containing an aqueous solution of MB (3 mL, 7.5 ppm) and NaBH<sub>4</sub> (1 mL, 3.8 ppm) was prepared in a quartz cuvette. The nanofiber catalyst (6.0 mg), PAN or PAN/zeolite, was added to an aqueous solution of MB and NaBH<sub>4</sub>. The smartphone-based spectrometer system was designed for screening the reaction kinetics by measuring their time-dependent absorption spectra. Hence at certain time intervals, the spectral images of the dye solutions were taken. The performance of the smartphone spectrometer was compared with that of a calibrated spectrometer.

## 3 Results and discussion

## 3.1 MB absorption studies

We first investigated the smartphone-based spectrometer system for measuring the absorbance of MB solutions at different concentrations. The concentration range was adjusted according to the sensitivity of the smartphone. MB solutions were prepared as 0.1, 0.5, 1.0, 2.5, 5.0, 7.5 and 10.0 ppm. Fig. 2a shows the color change of MB solutions from low to high concentration. The color of the solutions becomes darker gradually with increasing concentration of MB. Fig. 2a also demonstrates the spectral images of the serial MB solutions taken by the smartphone camera using the 0.25 mm diameter fiber. Since the characteristic absorption band of MB is around 664 nm, which corresponds to the red color, we particularly paid attention to observing the color at the end of the whole visible spectrum during the experiment. Accordingly, the blue and green spectra were saturated due to the non-linear responsivity of the smartphone camera to wavelength. To obtain the absorbance spectrum, the multicolored images were processed and the value of absorbance (A) was calculated using the Beer-Lambert law,

$$A = \log_{10} \frac{I_0}{I} \tag{1}$$

where  $I_0$  is the reference transmitted light intensity of distilled water (DW) and *I* is the transmitted light intensity of the solutions. Fig. 2b shows the typical spectrum obtained from the smartphone spectrometer compared to that from the commercial spectrometer. For low concentrations of MB (0.1 ppm to 5.0 ppm), both spectra are similar around 664 nm, while they are separated at high concentrations. The calibration curves for the MB solutions measured by the smartphone and commercial spectrometers are shown in Fig. 2c, with the regression coefficients of 0.998 and 0.997, respectively. Even though they both present perfect linear relationships, there is a small shift in absorptivity. This shift was reported before<sup>10,25</sup> as due to internal filters of the smartphone camera and the non-linear responsivity of the sensor. This shift also suggests that the absorptivity of the studied analyte should be studied using the same smartphone. The limit of detection (LOD) was calculated as 0.5 ppm which is three standard deviations above the distilled water measurement.<sup>26</sup> The sensitivity of the smartphone spectrometer system was found to be 0.1817 ppm<sup>-1</sup> that is also the slope of the fitted absorption line (RAW) given in Fig. 2c. The dynamic range of the sensing system is 0.5–10 ppm.

To test the efficiency of the JPEG image format in absorbance measurements, images captured for different concentration values were processed. As illustrated in Fig. 2c, the absorbance values obtained from spectral images in JPEG format show a non-linear relationship fitted to a seconddegree polynomial as opposed to the RAW format. Moreover, due to image post-processing during JPEG image reconstruction, the data spectrum at 664 nm for 10 ppm concentration shows zero transmission, and hence fails to supply an absorbance value.

## 3.2 Characterization of PAN/zeolite composite nanofibrous membranes

The SEM micrographs of zeolite powder, and nanofibers of PAN and PAN/zeolite are shown in Fig. 3a-c. The zeolite powder was composed of nanosized zeolite particles. The PAN nanofibers were smooth and uniform with an average diameter of 340  $\pm$ 50 nm. The diameter of the PAN/zeolite nanofibers increased to 550  $\pm$  95 nm due to the addition of zeolite into the PAN solution. Zeolite particles were uniformly distributed without any aggregation along the fibrous membrane. Furthermore, the energy dispersive X-ray spectrometer (EDS) spectrum shows oxygen, silicon, and aluminum signals for the PAN/zeolite nanofibers (see Fig. S2, ESI†). The distribution of the additional elements in the mapping image indicates the successful incorporation of the zeolites into the nanofibers. The inset images in Fig. 3b and c show the CAs of the PAN and PAN/ zeolite composite nanofibers. The PAN fibrous membrane showed a CA around 92°, whereas the composite membranes with 10 wt% zeolite content showed a CA around 86°. The decrease in the CA of the composite nanofibrous mat is due to the hydrophilic nature of the zeolites.<sup>27</sup> The FTIR spectra of the nanofibers of PAN and PAN/zeolite, as well as the zeolite powder are presented in Fig. 3d. The broad bands in the range of 3000-3600 cm<sup>-1</sup> are assigned to the water and hydroxyl groups. The corresponding bending vibration of water molecules occurs at 1665 cm<sup>-1</sup>. The band at 1020 cm<sup>-1</sup> is attributed to the asymmetric stretching of SiO<sub>4</sub> and AlO<sub>4</sub> tetrahedra for the zeolite.28 A characteristic band of nitrile groups in the PAN chain at 2240 cm<sup>-1</sup> is present on the surface of both nanofibers. The absorption bands observed at 2930 and 1460 cm<sup>-1</sup> are due to the C-H bonds. In the spectra of composite nanofibers, a new absorption band appeared at 1040  $\rm cm^{-1}$  supporting the successful zeolite modification.

## 3.3 Adsorption of MB by electrospun nanofibers

The catalytic activity of the PAN/zeolite composite nanofibers was tested in a model reaction taking place between MB dye and the reducing agent NaBH<sub>4</sub>. The reduction of MB occurs in the presence of a catalyst even if there is a reducing agent in the reaction medium.29,30 In order to understand the catalytic activity of the PAN/zeolite composite nanofibers, this reaction was selected for observation. The intensity of the MB absorption band at 664 nm was again followed by measuring the optical density of the solution. Fig. 4a shows the variations in the absorbance spectra of MB solution in the presence of PAN and PAN/zeolite nanofibers. There is an initial drop in MB absorbance due to addition of NaBH<sub>4</sub>. In the case of PAN nanofibers, the absorbance stays unchanged for the duration of 12 minutes. On the other hand, using PAN/zeolite nanofibers, the characteristic absorption peak of MB decreases as a function of time corresponding to a concentration reduction from the initial value of 6.45  $\pm$  0.38 ppm to 1.72  $\pm$  0.14 ppm. Inset spectrograms show the images taken from the initial MB solution and reduced MB solution after adsorption by using PAN/zeolite nanofibers.

Fig. 4b illustrates the photographs of the MB solutions containing PAN or PAN/zeolite nanofibers after 15 minutes. In line with these results, the bright blue color of MB turned a lighter color indicating the consumption of MB in the medium by the redox reaction. The blue color of PAN/zeolite nanofibers is also a clear sign of the physical adsorption of the dye on the nanofibrous mat. The color of the MB solution containing PAN nanofibers showed almost no change.

## 4 Conclusion

In this paper, we have reported an ultra-low cost, portable, plastic fiber based smartphone spectrometer platform for the investigation of MB dye adsorption using electrospun PAN/ zeolite composite nanofibers. We demonstrated the effect of fiber size on the spatial and spectral resolution. We processed color spectrum images in RAW and JPEG formats, showed that the RAW format is preferred for absorbance experiments, and obtained a dynamic range of 0.5 to 10 ppm. The sensing platform was able to detect down to 0.5 ppm, with a sensitivity of 0.1817 ppm<sup>-1</sup>. The PAN/zeolite nanofibers showed higher catalytic activity than those of PAN due to the presence of zeolite nanoparticles having high ion exchange capacity and enhanced surface area. The results of this study indicate that the proposed smartphone spectrometer is capable of replacing benchtop spectrometers for field-deployable instrumentation for environmental sustainability. The cost of the proposed cradle could be further decreased by using an alternative manufacturing method, such as injection molding. We envisage this strategy for numerous applications such as water treatment management, point-of-care sensing, performance and lifestyle monitoring.

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