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Colorimetric analysis of water and sand samples performed on a mobile phone

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ABSTRACT

Analysis of water and sand samples was done by reflectance measurements using a mobile phone. The phone's screen served as light source and front view camera as detector. Reflected intensities for white, red, green and blue colors were used to do principal component analysis for classification of several compounds and their concentrations in water. Analyses of colored solutions and colorimetric reactions based on widely available chemicals were performed. Classification of iron(III), chromium(VI) and sodium salt of humic acid was observed using reflected intensities from blue and green light for concentrations 2–10 mg/l. Addition of complex forming sodium salt of ethylenediaminetetraacidic acid enabled the discrimination of Cu(II) ions in the 2–10 mg/l concentration range based on reflection of red light. An alternate method using test strips for copper solutions with the phone as reader also demonstrated a detection limit of 2 mg/l. Analysis of As(III) from 25 to 400 μg/l based on reflection of red light was performed utilizing the bleaching reaction of tincture of iodine containing starch. Enhanced sensitivity to low concentrations of arsenic was obtained by including reflected intensities from white light in the analysis. Model colored sand samples representing discoloration caused by the presence of arsenic in groundwater were analyzed as a complementary method for arsenic detection.

1. Introduction

Access to clean and safe drinking water is a prerequisite for leading a healthy life. The UN's Millennium Development Program recognizes this fact by establishing the goal of halving, by 2015, the proportion of people lacking access to safe drinking water and basic sanitation. The problem is especially acute for people living in rural areas with drinking water supplies coming from unmonitored streams and wells where local and regional conditions determine water quality.

South and Southeast Asia are densely populated areas where water supplies are from major river basins and groundwater wells. Recurring problems with pathogens in rural surface waters have led governments to encourage use of groundwater supplies. While this has reduced the outbreak of waterborne diseases it has led to consumption of water high in metal contamination which poses a long term health hazard to the populace. Biogeochemical–hydrological processes regulate the concentrations of metals in groundwater and the most dangerous component is arsenic [1,2]. Prolonged consumption of water having high arsenic levels has been shown to lead to increased incidence of cancer and cardiovascular disease. Weathering of rocks containing sulfide minerals in the Himalayas leads to arsenic accumulation in iron oxyhydroxides with the final step being microbial reduction of the minerals releasing As(III) into the groundwater. Thus the presence of other metals in water supplies such as iron and manganese can confirm the occurrence of arsenic [2]. The danger from arsenic in groundwater is not limited to drinking water but also from irrigation water leading to accumulation in soil and crops [3].

Although arsenic contamination of ground water supplies is a world wide problem it is particularly acute in South Asia because of contaminated surface water. In addition, rural populations are poorly equipped for conducting water analysis of individual wells. Depending on depth, the number of wells in Bangladesh and West Bengal in India having arsenic concentrations which exceed the WHO guideline of 10 μg/l is more than 50%. Concentrations up to 1000 μg/l are not uncommon [2]. The sheer number of wells makes monitoring by government agencies impossible and the availability of analysis equipment to consumers is limited.

An analysis system based on readily available equipment and chemicals is an attractive starting point for empowering local populations to ensure the quality of their drinking water. Social factors have been shown to be important for acceptance of mitigation efforts and use of safer deep wells in the region. These are often related to support from family and neighbors with respect to the need for improving the quality of the local drinking water supply [4]. The social situation of women should also be a consideration with respect to analysis methods since their familiarity with the techniques employed will accelerate lifting the awareness of the community to work for safer water supplies for their families.

Chemical analysis using consumer electronics such as computer screens and web cameras [5–7], optical disc drivers [8,9] and

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flat bed scanners [10] has been introduced as a readily available analytical technique. A current generation mobile phone also has all required capabilities for optical analysis. Use of mobile phone cameras as a spectroscopic sensor [11] and to record images in telemedicine applications [12,13] have been reported. We describe here the use of a Nokia 6220 classic mobile phone to perform analysis of water samples containing a variety of substances found in surface and ground waters. Using the phone's screen as light source and secondary front view camera as detector made possible reflectance measurements from sample surfaces. The samples chosen demonstrate how the phone can be used for home analysis of water containing colored compounds and substances which can be colorimetrically analyzed using widely available chemicals. Use of the technique to monitor sand samples in connection with establishment of wells is also described.

2. Experimental

A Nokia 6220 classic mobile phone was used as both light source and detector in the study. Its operation for colorimetric analysis has been described previously [14]. The screen served as a controlled light source and the front view camera acquired spectral information in the form of images. The 6220's screen is 34 mm wide and 46 mm long and has 24 bits color resolution in TFT display with 320 × 240 pixels in QVGA format.

The main 5 megapixel camera is situated on the back side of the phone and would be first choice as detector in a two camera transmission measurement system. The secondary front view camera was used for the single phone reflectance measurements of this study. It is designed for video calls and has both CIF 352 × 288 pixels for image detection and QCIF 176 × 144 pixels for video recording. The front camera's effective focal length was determined to be 70 mm. At that distance video frames of 37 mm × 45 mm filled the entire 176 × 144 pixel field of view of the camera.

Software was developed for phone operation in an automatic exposure mode. NetBeans open source Integrated Design Environment was used to design, test and implement software in the form of a MIDLet (Mobile Information Device applet), which controlled the screen as light source and simultaneously acquired images to obtain reflectance intensities from the samples. A key feature of the software was overriding the phone's JSR-234 (Java Specification Request) generated security warning messages which required a yes click in order to proceed further. Fixed length videos were recorded by the camera by clicking yes to all four security messages before the video capturing started. Recording was done at 15 frames/s with each color taking 6.8 s of display time. Since the video capturing rate was faster than the screen illumination rate, several frames were captured and saved for each illumination color. Consecutive series of 50 frames were selected and processed for each color. Data was stored .3gp files using H.263 video compression technology.

The front camera's CMOS technology based sensor recorded reflectance intensities from the samples in the form of 176 × 144 pixel color images. AVS video converter6 was used to convert the .3gp mobile phone videos into image format such that each frame of the parent video file would have the same size in terms of pixels after conversion into Bitmap (bmp) image format. These bmp images files were loaded into MATLAB-R2007b for standard function processing. The processing consisted of image data import (imread), concatenation of the 50 frames/color (strcat), grayscale conversion (rgb2gray), scaling image data to the full range (imagesc), error estimation (std), and finally computation of reflectance intensity values corresponding to each pixel of the camera sensor (mean). Error estimation was used to guide development of the measurement set-up with the goal of obtaining standard deviations for reflected intensities <1%.

The measurement cell was a weighing boat of white polystyrene having rounded corners and dimensions 80 mm × 80 mm × 25 mm from VWR International. A black laminated paper was positioned under the boat and the phone was placed on a mechanical stand which allowed alignment between samples and the screen/camera. A plastic box was placed over the setup to exclude ambient light during measurements. Solutions of the following substances (all from Sigma–Aldrich) were prepared in deionized water: sodium salt of humic acid, iron(III)chloride hexahydrate, potassium dichromate and sodium arsenite. Stock solutions were diluted to obtain concentration series of the compounds. The disodium salt of ethylenediaminetetraacetic acid dihydrate (EDTA) was dissolved in deionized water for complexation with Cu(II) at a 1:1 molar ratio. USP tincture of iodine was prepared by dissolving 0.2 g iodine and 0.24 g sodium iodide in 5 ml ethanol + 5 ml deionized water. Starch solutions were prepared by heating 1 g starch in 100 ml deionized water at 80 °C. Reflectoquant test strips for copper were from Merck. Volume of solutions in the measurement cell was 70 ml. Model colored sand samples were prepared by adding 7 ml of black/orange dye solutions to 30 g of sand with drying at room temperature. Reflected intensities for white, red, green and blue illuminations on the screen were recorded during randomized measurement series having up to 36 samples with deionized water references included at intervals.

The reflected intensities were used to classify samples using principal component analysis (PCA) [15]. This enabled us to evaluate how well the analysis system differentiated between different substances in the water, their concentrations and the colors contributing to the compound discrimination. The program Sirius from Pattern Recognition Systems AS (Oslo, Norway) was employed to obtain score plots describing classification of samples based on reflected intensities from selected illumination colors.

3. Results and discussion

In order to perform the measurements using a single mobile phone the front view camera was employed as detector. This resulted in reflectance from sample surfaces being the basis of the analyses. Colorimetric analysis of liquid samples usually employs transmittance measurements and reflection is an alternative when solutions exhibit turbidity [16–18]. Reflectance is, however, used extensively for distance monitoring of lakes and oceans. Diffuse reflection of light from a water surface determines its color [19] and can be used to identify the particles, chemicals and life forms present [20,21]. Light entering the water interacts with molecules and particles by both inelastic scattering and adsorption of photons [22,23]. The strategy in this investigation was thus to compare the spectral distribution of reflected light from deionized water reference samples [24] with samples containing specific contaminants.

The 70 mm focal length limited light reflected to the camera to only the surface and subsurface of the liquid samples. The analogy with ocean reflection is thus for the case of deep water with little contribution to reflection from the bottom [25]. The grayscale image light reflected from 70 ml of deionized water in the sample vessel during white light illumination is shown in Fig. 1. Because of the asymmetric screen/sample/camera configuration there was non-uniform illumination of the sample with respect to the camera's field of view (see supplementary Fig. 1 for description of the measurement set up). The dark square region at the bottom of Fig. 1 is the area of the sample receiving the highest intensity illumination from the screen resulting in saturation of the camera for reflected light from that area. A faint outline of the water level in the vessel can be seen along horizontal row 10 and vertical column 150. A

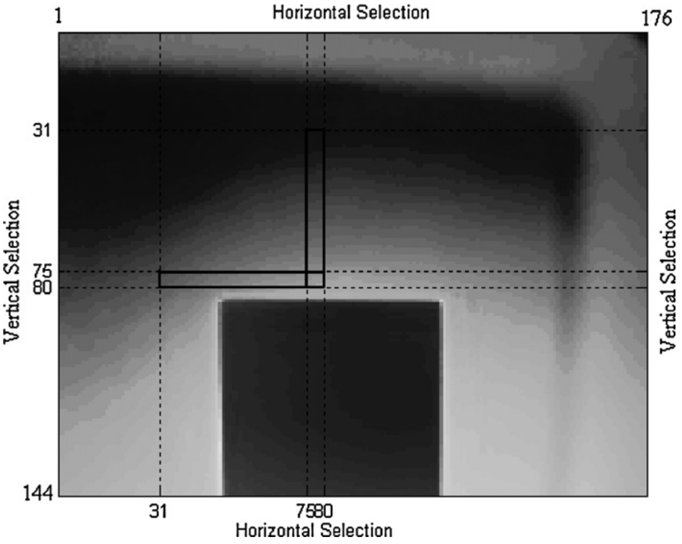

Fig. 1. Pixel grid scheme for reflected light to camera as grayscale image illustrated for white light reflected from 70 ml deionized water.

survey of the intensities from the pixels above the dark square region indicated that pixels in rows 75–80 and in columns 75–80 (pixels 31–80 in both directions as indicated by the dark solid lines in Fig. 1) contained the most useful information for analysis. We used 50 pixels each from rows and columns 75 and 80 for the illuminating colors white, red, green and blue ($2 \times 4 \times 100 = 800$ intensities for each sample) to illustrate operation of the phone for colorimetric analysis.

Reflected intensities for the solutions made from the selected compounds were recorded for the four illuminations. PCA treatment of the data yielded classifications of the different solutions and concentrations. Fig. 2 shows the results for three different colored compounds, sodium salt of humic acid (brown in the solid state), iron(III)chloride hexahydrate (yellow) and potassium dichromate (orange). PCA based on reflected intensities from green illumination resulted in some classification of concentrations for humic acid and iron(III) but not of the compounds as shown in the left-hand plot of Fig. 2. Compound classification improved when reflected intensities from two illuminations were used in the analysis with green and blue giving the sharpest discrimination, even for the lowest concentrations relative to the deionized water references, as shown in the right-hand plot. Solutions with increasing concentrations of the compounds extended out along different projections from the references.

Since green and blue illuminations were found to give the sharpest discrimination among the three compounds, an investigation was made of cyan illumination. It is the additive secondary color containing green and blue wavelengths in the RGB color system and yields a spectral radiance equivalent to green + blue on the phone's screen [14]. Thus as illuminating color it should be suitable for discrimination of the three compounds. A score plot obtained using reflected intensities from only cyan illumination, however, yielded only a concentration discrimination of the solutions similar to what is shown in the left-hand plot of Fig. 2. Compound discrimination was observed when pixel intensities from cyan illumination was combined with green or blue illumination but in both cases the separation of compounds and concentrations was not as sharp as is shown in the right-hand plot of Fig. 2. The lack of equivalence between cyan and green and blue illumination in discriminating the samples lies in the different illumination intensities of the colors from the screen [14]. Illumination intensity ratios for RGB channels are roughly 5:9:3, which is why reflected intensities from green illumination were highest among the colors. This is illustrated in Fig. 3 where intensities for row and column 75 are plotted for several colors and samples. Since the blue component of the cyan illumination contributed very little to the total incident light intensity, the reflected intensity was dominated by the green component. A complementary discrimination of the samples was observed for the reflected intensities from the individual green and blue illuminations. Green illumination discriminated between chromium/deionized water and iron/humic acid while blue illumination separated chromium/humic acid from iron/deionized water. This synergy between the two colors for analyzing the samples was lost during the cyan illumination.

Although the purpose of the study was not to produce a finished analytical system, both the potential and the weakness of the method are illustrated in Fig. 2. Colored liquid samples could be classified both according to identity and concentration of dissolved substances. There is a potential for compiling a library of optical patterns for a number of compounds relative to a reference standard such as deionized water which could be used to identify unknowns. However, to obtain solutions displaying sufficient coloring the concentrations of species such as metal ions must be quite high. The low degree of explanation for PC2 in the right-hand of Fig. 2 also indicates the low selectivity of the method. The cam-

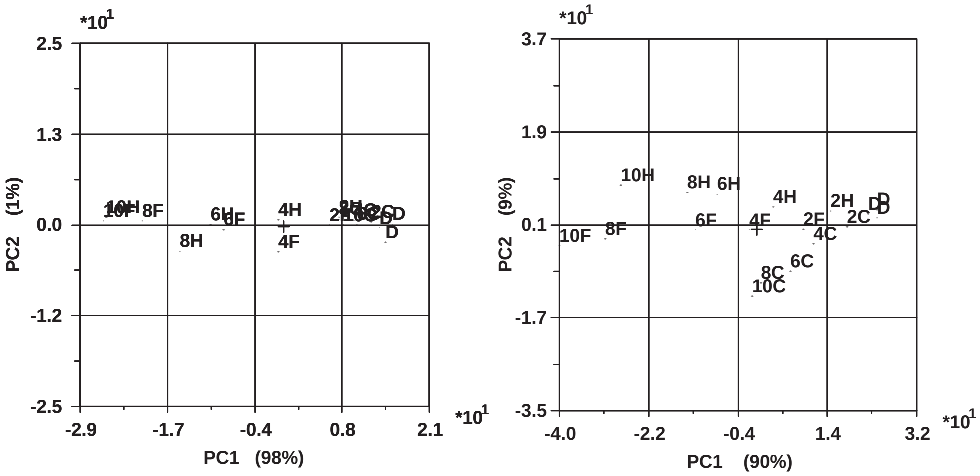

Fig. 2. Score plots for solutions of sodium salt of humic acid (H), iron(III)chloride (F) and potassium dichromate (C) when reflected intensities for green illumination, left, and green and blue illuminations, right, from rows and columns 75 and 80 were used in the PCA. Solution concentrations are indicated as mg/l metal ion or acid and three reference deionized water samples, D, are included.

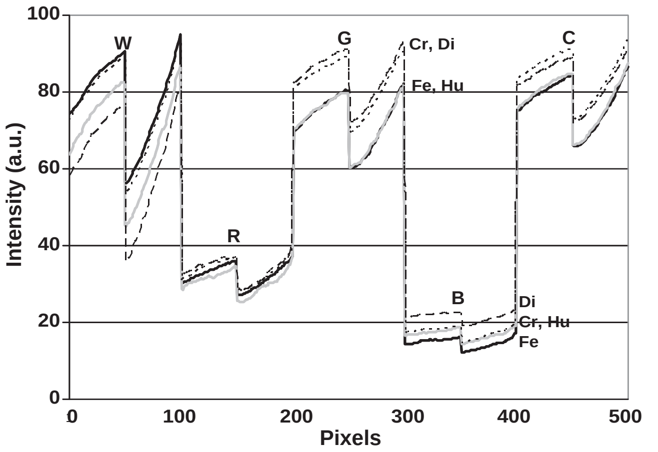

Fig. 3. Reflected intensities for row and column 75 for white (W), red (R), green (G), blue (B) and cyan (C) illuminations for the 10 mg/l samples of iron(III)chloride (solid line, Fe), potassium dichromate (short dashed, Cr), sodium salt of humic acid (gray, Hu) and deionized water (long dashed, Di). Large letters indicating colors are placed between the pixels representing row and column.

era is constructed with inexpensive consumer electronics based on CMOS technology and the screen's 24 bit color capability provides only 8 bit resolution in each color channel. In addition, the screen's illumination intensity is unevenly distributed among the colors. However, despite these built-in limitations, the system was able to quite well discriminate among three compounds which all exhibited similar yellowish–brownish colors in solution.

Both the sensitivity and the selectivity could be improved by employing complexing reagents. The detection limit of Cr(VI) in distilled water has, for example, been reported at about 0.3 mg/l using absorption spectroscopy with path length 10 mm [26]. Much lower detection limits for Cr(VI), however, have been demonstrated using reaction with diphenylcarbizide at low pH to form colored complexes with absorption maximum at about 540 nm. The complexation reaction has even enabled hexavalent chromium detection at the World Health Organization maximum recommended concentration level of 0.05 mg/l using computer screen assisted technology [7]. A number of such complexation reagents would both improve the detection limit of the mobile phone method and increase the variety of colors available for identifying dissolved substances.

An example of enhancing the coloration of solutions is addition of the widely used complexing agent EDTA to copper solutions. EDTA is a component of such products as detergents and cosmetics because of its metal complexing property. It is being gradually replaced by biodegradable S,S′-ethylenediaminedisuccinic acid but still finds application in shampoos where $CuEDTA^{2-}$ acts as blue colorant. The use of this colored complex for Cu analysis is demonstrated in the left-hand plot of Fig. 4. Classification of copper(II)chloride solutions was obtained when light absorption in the 600–800 nm region was increased by a factor of about 6 relative to pure copper solutions, as determined by transmission absorption spectroscopy, upon formation of $CuEDTA^{2-}$ complexes. Since solution color even after complex formation was still quite weak compared to the compounds analyzed in Fig. 2, an average of reflected intensities for pixels in the horizontal rows 75 and 80 from four measurements on the samples was used in the analysis.

An alternative method for water analysis would be use of test strips. These are available for a number of metal ions. Copper(II) analysis is based on reduction to Cu(I) followed by reaction with 2,2′-biquinoline to form a violet complex [27]. Reflectometric determination of concentration is done in a dedicated reader with detection limit at 5 mg/l Cu. Copper(II)chloride solutions were prepared and results for concentration classification is shown in the right-hand plot of Fig. 4. The 10 mg/l Cu solution gave a light pink color to the test strip. The weak coloration combined with the heterogeneous surface of the strip resulted in considerable scatter in the reflected light intensities recorded at the individual camera pixels. A trial and error evaluation of the illumination and test strip geometric parameters led to the conclusion that an average from three measurements of the mean of 10 × 10 pixel intensities at the center of the strip for red and green illumination yielded the best discrimination of the Cu concentrations. As shown in Fig. 4 the color intensity on the strip was first discriminated at the 2 mg/l Cu concentration which is in line with the detection limit available using the strip reader. While the cost of commercially available test strips would be limiting for this application of the phone, less expensive paper based indicators have been reported [28,29]. Recording of images by the back camera in ambient light resulted in poor discrimination of the test strip colorations.

Redox reactions using readily available chemicals can also be utilized to analyze for metals in solution. This is shown in Fig. 5 where addition of tincture of iodine and starch to solutions containing As(III) ions resulted in a bleaching of the blue indicator color as

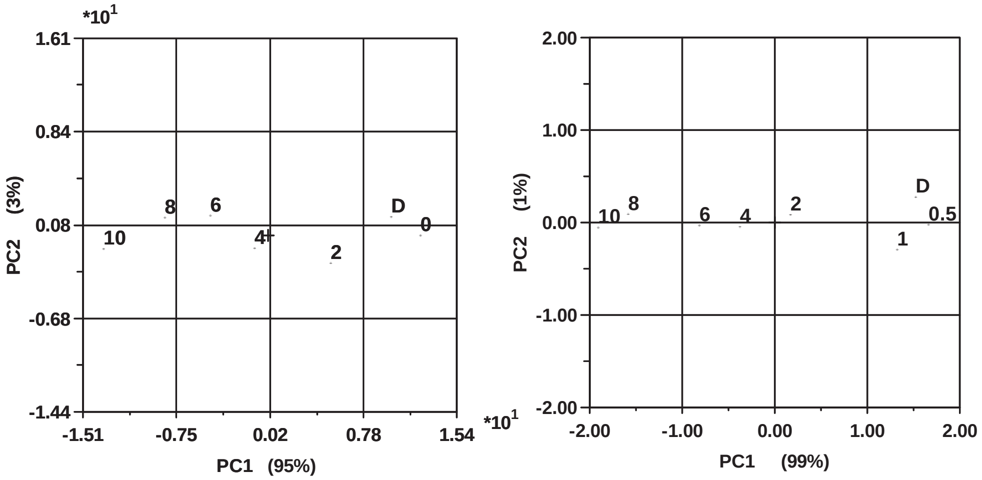

Fig. 4. Score plot for copper(II)chloride solutions with added Na_2EDTA when averages from four measurements of reflected intensities from red illumination for horizontal rows 75 and 80 were used in the PCA, left. Score plot for pure copper(II)chloride solutions when the mean of 10 × 10 reflected intensities for the center of test strips, averaged for three separate measurements, from red and green illuminations were used in the PCA, right. Cu(II) concentrations are indicated as mg/l. Samples containing only deionized water are labeled D.

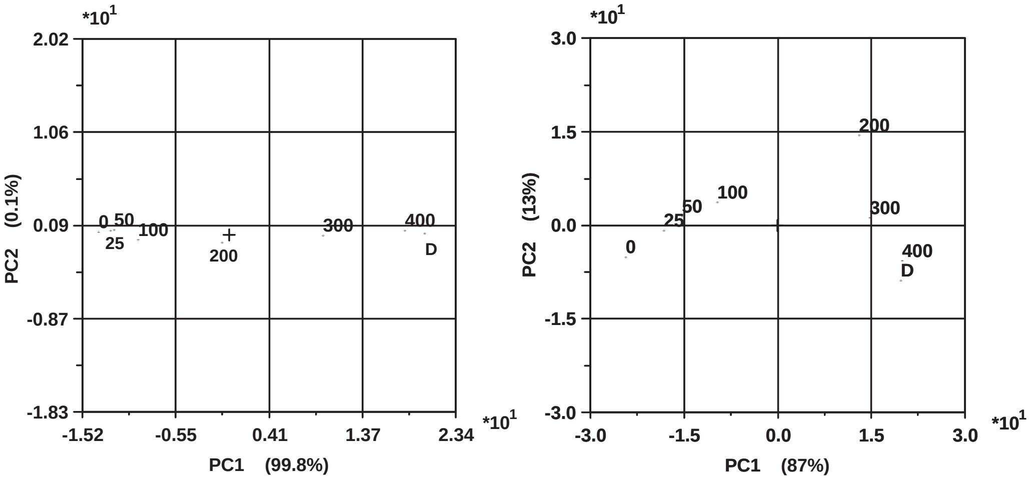

Fig. 5. Score plots of sodium arsenite solutions after reaction with tincture of iodine containing starch when reflected intensities for rows and columns 75 and 80 from red illumination, left, and red and white illuminations, right, were used in the PCA. As(III) concentrations are indicated as μg/l. Samples containing only deionized water are labeled D. All non-D samples contained 0.5 ml starch solution and 0.03 ml tincture of iodine.

iodine oxidized As(III) to As(V) which disrupted the iodine–starch complex [30]. Fig. 5 shows the discrimination of As(III) samples for concentrations up to 400 μg/l following the bleaching reaction. Red illumination yielded a good discrimination of the concentrations with high degree of explanation along PC1. An enhancement of the discrimination at low concentrations was observed when reflected intensities from both red and white illuminations were used together as variables in the PCA as shown in the right-hand plot of the figure. Low and high concentration projections were obtained and an increase in degree of explanation for PC2 enabled a clearer discrimination of the lower concentrations. The enhancement of the lower concentration discrimination by white illumination can be explained by observing plots similar to Fig. 3. Curves for the reflected intensities from the white illumination were clearly separated for the lower concentrations relative to the curves for blue and green illuminations.

It has recently been shown that the color of aquifer sands can be used to indicate the presence of As in groundwater [2]. Orange colored sands indicate the presence of Fe(III)oxides which have been shown to be related to low As content in water. Sands colored gray by reduced iron oxides give an indication of the possible occurrence of higher As concentrations. The phone device was also tested for analyzing the coloring of solid sand samples. Since the volume of the 30 g samples was only about 19 ml the position of the measurement vessel had to be raised in order to maintain the 70 mm focal length of the camera. A much more heterogenous image was observed as compared with what was obtained from liquid samples as shown in Fig. 1 (reflected illumination from sand shown in supplementary Fig. 2). Fig. 6 shows the classification of the model orange and grayish colored sands. The more heterogeneous nature of the recorded images is reflected in the classifications obtained. Only intensities from red illumination yielded reasonable color classifications. A trial and error evaluation of the reflected intensities indicated that the horizontal pixels in row 80 gave the best sample classification with a relatively high degree of explanation along the PC1 projection.

4. Conclusions

Use of a mobile phone for colorimetric analysis of liquid and solid samples has been demonstrated. Diffuse reflection of light from samples illuminated by the phone's screen yielded information about identity and concentration of compounds in liquid samples. Compounds giving visibly colored solutions could be analyzed directly by the phone. The phone's screen does not provide equal illumination intensities for different colors. Inexpensive CMOS electronics in the camera limit detection resolution. Despite these drawbacks the system has the capability to discriminate between colored solutions having very similar optical properties. Solution colors can be enhanced by employing complexing chemicals which could also extend the range of colors available for analyses. Reactions with colored indicator solutions provide a means to utilize the system's colorimetric analysis capabilities even for colorless compounds. In this area the mixing of reflected intensities from different illumination colors demonstrates the possibility for enhanced sensitivity to low concentrations of arsenic compounds.

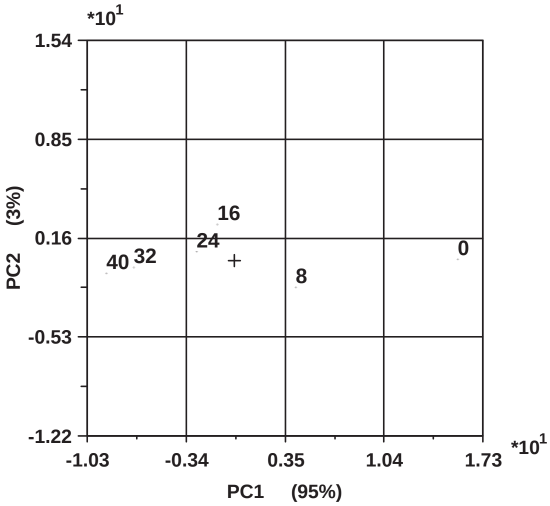

Fig. 6. Score plot of model colored sand samples when reflected intensities from red illumination for horizontal rows 75 and 80 were used in the PCA. Sample designations refer to volume % black dye solution contained in the orange dye solution used for the coloring.

Solid samples can also be analyzed using this technique. Solid surfaces are more heterogeneous than liquids which increases the difficulty of colorimetric analysis by reflected light. The phone can be used to replace a dedicated reader for analyses using test strips with the controlled illumination from the screen enhancing sensitivity. Discoloration of sand indicates the presence of chemicals such as arsenic in water and discrimination of the sand coloring would be aided by use of the phone analysis method relative to visual observations.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.talanta.2011.03.016.

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