



## Inkjet printed polyelectrolyte patterns for analyte separation on inherently porous microfluidic analytical designs

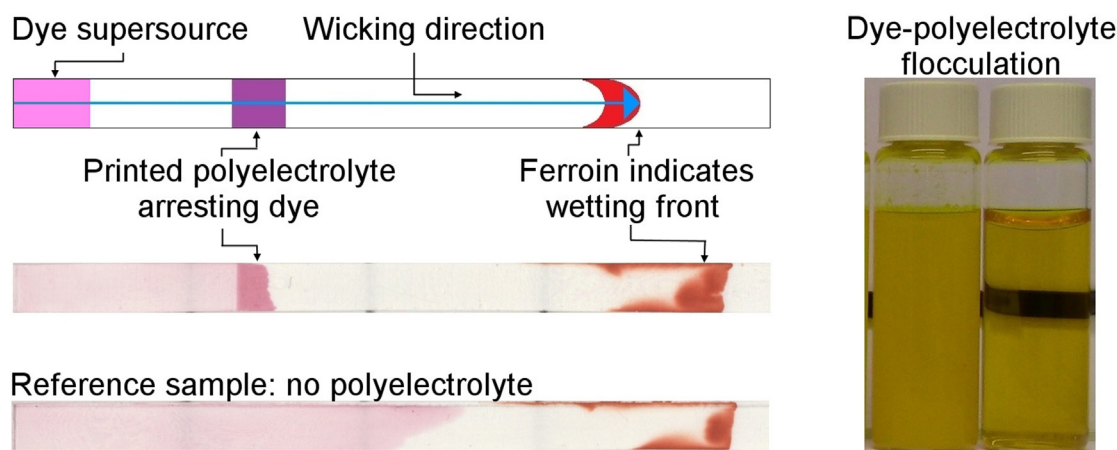


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### GRAPHICAL ABSTRACT



### HIGHLIGHTS

- Custom porous pigment material in coating transporting liquid by capillary action.
- Anionic and cationic polyelectrolyte regions applied by inkjet printing.
- Anionic colourants separated from solution at cationic polyelectrolyte regions.
- Electrostatic charge differences drive separation.
- Separation mechanism could be used to enhance analytical/diagnostic applications.

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### ABSTRACT

Analytical devices made of inherently porous material can provide platforms for sensor applications in point-of-care medical diagnostics and environmental monitoring. However, separation and concentration of analytes on such devices has received limited attention. Neither have porous coatings, with their potential high surface area for chromatographic separation, in such devices been studied in detail. This study investigates inkjet printed polyelectrolyte patterns on such a coating as a possible method for concentration and separation of cationic and anionic compounds through surface charge interaction.

**Abbreviations:** FCC, functionalised calcium carbonate; MFC, micro-fibrillated cellulose; PolyDADMAC, poly(diallyldimethylammonium chloride); NaPA, sodium polyacrylate; SEM, scanning electron microscope; TLC, thin layer chromatography.

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Both anionic (sodium polyacrylate) and cationic (poly(diallyldimethylammonium chloride)) polyelectrolytes were printed on a custom designed porous pigment coating, having fine internal particle pore structure to ensure high surface contact with analytical samples. Printed polyelectrolyte patterns were poorly visible under visible or ultraviolet light. In a proof of principle test, a controllable degree of separation of three anionic dyes (amaranth, tartrazine and uranine) from aqueous solution passing through a printed cationic polyelectrolyte region was observed. Separation of the two tested cationic dyes (crystal violet and methylene blue) on printed anionic polyelectrolyte regions could not be evaluated effectively with the set-up, since the dyes were too strongly arrested by the anionic micro-fibrillated cellulose binder in the pigment coating. The weakly cationic/zwitterionic dye rhodamine B was shown to remain free to pass in solution through either cationic or anionic printed polyelectrolyte regions. The principles illustrated can provide a basis for enhancing detection on certain analytical device designs.

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## 1. Introduction

Analytical devices made of paper can provide platforms for easily transportable, inexpensive and disposable sensors in various fields, including in-field environmental monitoring [1] and point-of-care medical diagnostics [2]. One research area of such devices is paperfluidics or paper-based microfluidics. On a paperfluidic device, aqueous liquid travels by capillary wicking without need for external pumping in a thin porous hydrophilic medium formed of cellulose-based fibres, where hydrophobic patterns can be applied to direct the flow.

Paperfluidics has further inspired study of devices based on other materials sharing certain properties with paper: thin planar form, permeable porous structure and hydrophilic surface chemistry facilitating capillary transport. Examples include speciality pigment coatings [3] and synthetic polymer nano-fibre mats [4]. Such materials provide a wider range of possibilities in regard to surface chemistry, pore matrix dimensions, reactive surface area, spatial resolution or thickness of the platform substrate. This emerging field could be called absorption-fluidics.

Liquid travelling on a paperfluidic or absorption-fluidic device may pass through reaction areas, where analytes of interest react with pre-applied reagents, before reaching a detection zone where the final result is analysed. Examples of demonstrated reactions include analysis of nitrite levels from saliva [5], detection of liver enzyme alanine aminotransferase from blood [6] and detection of glucose and ketones from urine [7]. Detection methods vary from colorimetric analysis, with the unaided eye or by external reader, to more sophisticated instrumental methods such as fluorescence microscopy or electrochemical detection [8].

Detection of analytes may be improved by separating undesired components from those of interest, or by differentially concentrating the analyte to obtain a stronger signal. Size-based separation on a paper pore matrix has been applied to agglutinated red blood cells in order to separate them from blood plasma, allowing colorimetric analysis of the plasma content [9]. Lateral flow tests, such as the commercial early pregnancy test for detection of human chorionic gonadotropin in urine, often use immobilisation methods to concentrate analytes of interest onto the detection zone [10]. On paper substrate analytes have been concentrated by evaporation in order to increase assay sensitivity [11], a mechanism benefiting from the well-known Marangoni flow “coffee-stain” effect to transport the analyte(s) to the perimeter of a drying liquid region [12]. Combination of electro-kinetic transport, local Nafion treatment and convergence of multiple channels has been demonstrated as a means for concentrating samples on chromatographic paper by ion concentration polarisation [13].

So far, the use of polyelectrolytes for separation or concentration on paper-based or absorption-fluidic analytical devices has been reported only on a few studies. Filter paper with polyelec-

trolyte multilayer surface structure has been demonstrated for concentration of viruses by electrostatic adsorption on a cationic polyelectrolyte top layer [14]. Two cationic polyelectrolytes, used as papermaking additives to improve retention and wet strength, have been studied for blood cell separation, with high molecular weight cationic polyacrylamide shown to increase retention of red blood cells on paper, presumably due to electrostatic interaction possibly assisted by some amount of cell flocculation [15]. Inkjet printed cationic polyvinylamine has been applied to sensor reaction areas to concentrate an anionic, yellow-coloured reaction product, 5-thio-2-nitrobenzoate [16] and ClBr-indigo dye [17].

This study investigates the use of inkjet printed polyelectrolytes to modify locally pore surface charge in order to separate and concentrate anionic and cationic compounds from liquid samples travelling by capillary action on an inherently porous, hydrophilic custom-designed high surface area particulate pigment coating. These polyelectrolytes are expected to adsorb initially on the pore walls and then in turn to interact with oppositely charged molecules. To this effect, two polyelectrolytes, cationic poly(diallyldimethylammonium chloride) (polyDADMAC) and anionic sodium polyacrylate (NaPA), were printed in aqueous solution on the coating. While polyelectrolytes can be added to a pigment coating slurry as additives, their local application by printing should keep reaction and detection zones free of them.

Both polyDADMAC and NaPA are found in industrial applications, e.g. as additives in papermaking, as dispersing agents in paper coatings and as flocculation supporting thickeners in wastewater purification. Besides their use as papermaking additives, polyelectrolytes have also been applied experimentally to papers as surface treatments, with multi-layer polyelectrolyte treatment found to control spreading of inkjet ink dyes on paper, with the specific effects depending on the number of layers and the nature of the top layer [18]. Also, the presence of cationic polyelectrolyte as additive in paper has been discovered to improve deinkability of papers printed with dye-based inkjet inks [19].

In order to form an effective printed polyelectrolyte separation or concentration zone, as investigated in this study, the following requirements should be met:

1. polyelectrolyte ink must be reliably inkjettable with sufficient dry solids content,
2. polyelectrolyte ink must penetrate the full depth of the porous substrate,
3. deposited polyelectrolyte shall not desorb from the substrate pore surfaces when re-wetted (a renowned problem if adsorption is not supported by charge (Coulombic) attraction with the surface or by van der Waals forces),
4. aqueous liquid with the analyte of interest is transportable within the porous substrate by capillary action,

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