Nanofabrication of electrochemical sensors for real-time monitoring of water quality from sensory evaluation: Taste Issues

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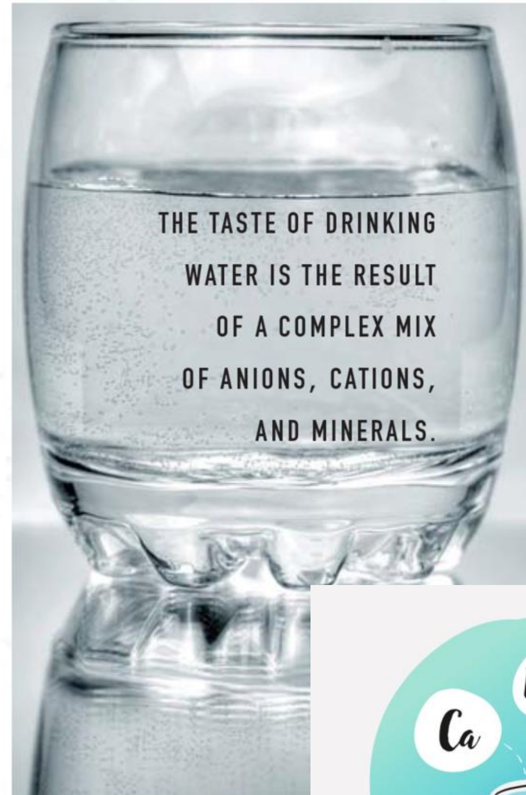
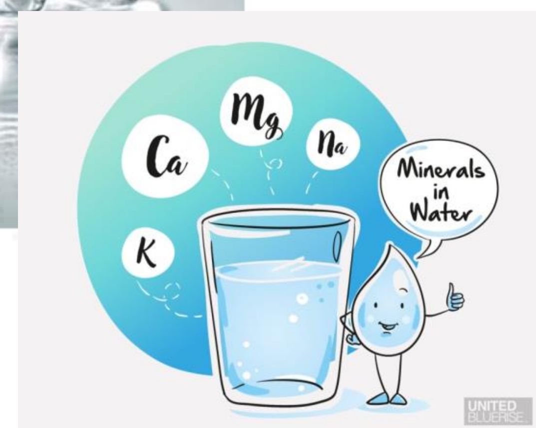
Background

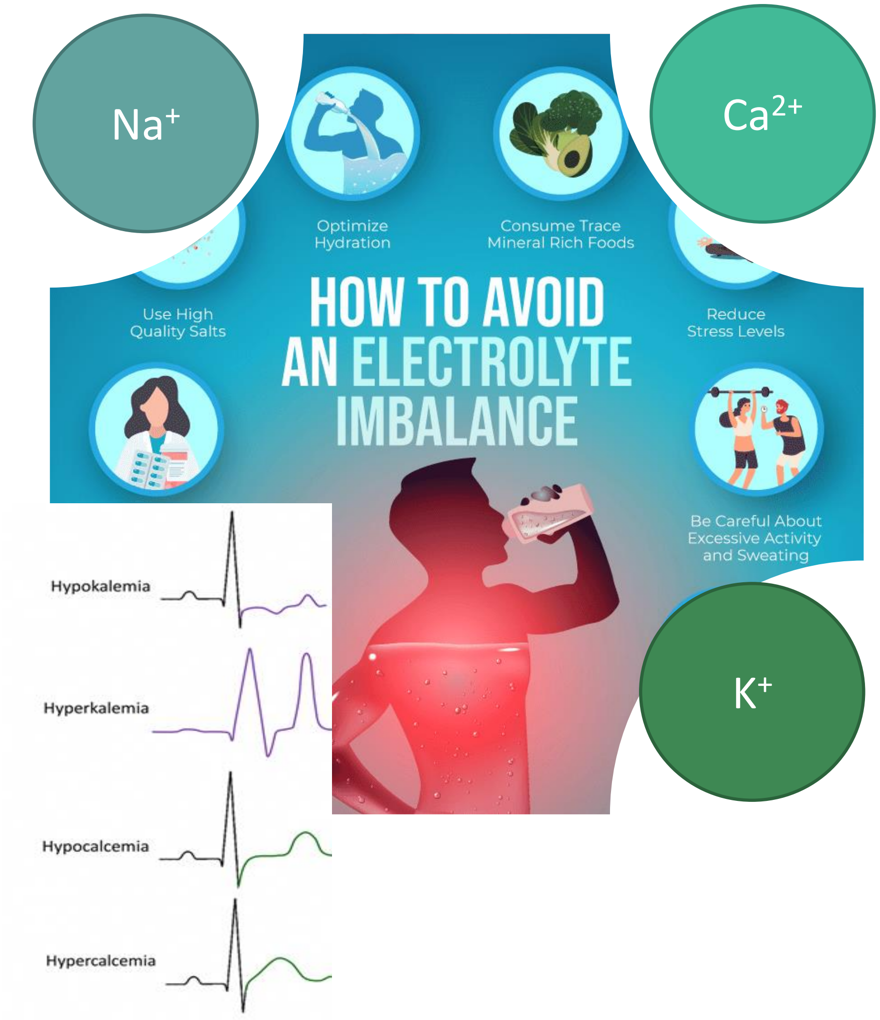
- Taste perception is critical for human life to be informed on nutrients uptake and avoid harmful substances.
- Human taste perception is able to identify a variety of different chemical substances in aqueous environment.
- Human sensory evaluation is typically used to obtain the taste information in food stuffs and beverages.
- Evaluation of taste quantization is limited to many application fields, therefore, there is a need for more objective and accurate detection methods compared to biological senses.





Background

- The concentrations of electrolytes including K^{+}, Na^{+} and Ca^{2+} are crucial in drinking water since low or high level uptake of these ions cause many diseases such as hypertension, cystic fibrosis, acute kidney injury, renal tubular acidosis.
- Development of miniaturized portable devices to detect electrolytes at the poin of need in water samples is crucial.



Background

- Water samples are classified according to taste descriptors such as sweet, sour, acidic, salty, bitter.

- Sourness comes from the presence of sodium whereas calcium ions in drinking water lead to bitterness. For example, the flavor is rated as 35% bitter, 32% sour, 29% sweet, and 4% salty at 1 mM of $CaCl_2$ solution. NaCl plays a crucial role in salty taste in drinking water.

- Ions in water samples is typically analyzed using spectroscopic techniques; however, these conventional methods require long analysis time, labor-intensive sample preparation steps, skilled laboratory personnel and expensive instruments.

The Aim of the study

- Electrochemical sensors are rapid, user-friendly, low-cost, portable and can be used without the need of time-consuming sample preparation at the point-of-need.
- Electrochemical signal changes is obtained by specific recognition and improving sensitivity is the crucial key to optimize the detection performance of taste-sensors.
- There are various sensitivity enhancement methods for taste-sensor based on receptor. For example modification of sensor surface using nanomaterials with high specific surface area and conductivity.
- In this study, electrochemical sensors modified with carbon based nanomaterials are developed for rapid, low-cost and highly sensitive detection of sodium, potassium and calcium ions. Also, a portable read-out devices is integrated with the nano-sensor array for simultaneous detection of these ions at the point-of-need [1-3].
- The performance of the electrochemical sensor array is evaluated by constructing a standard curve and conducting specificity analysis in water samples.

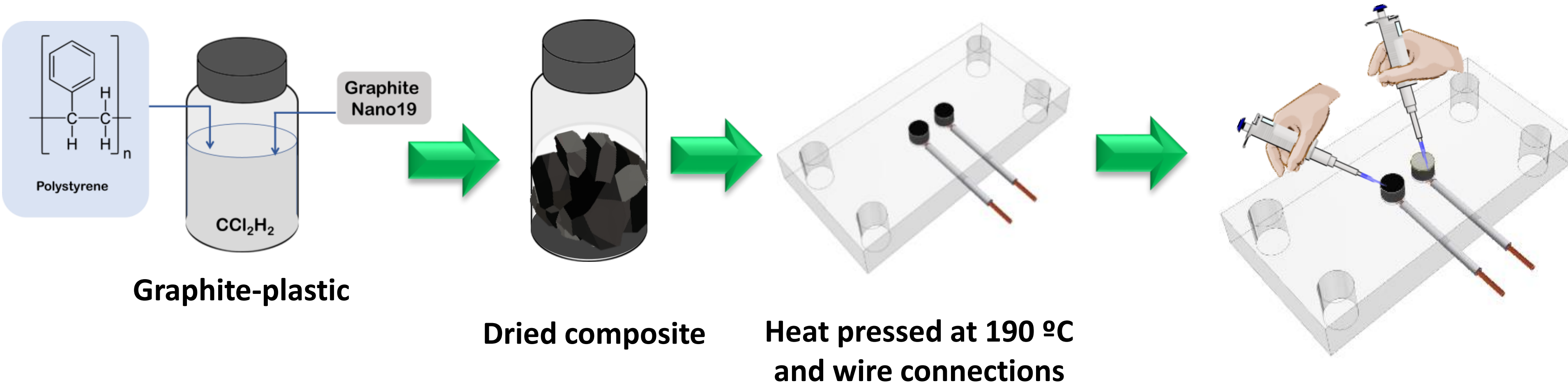
References

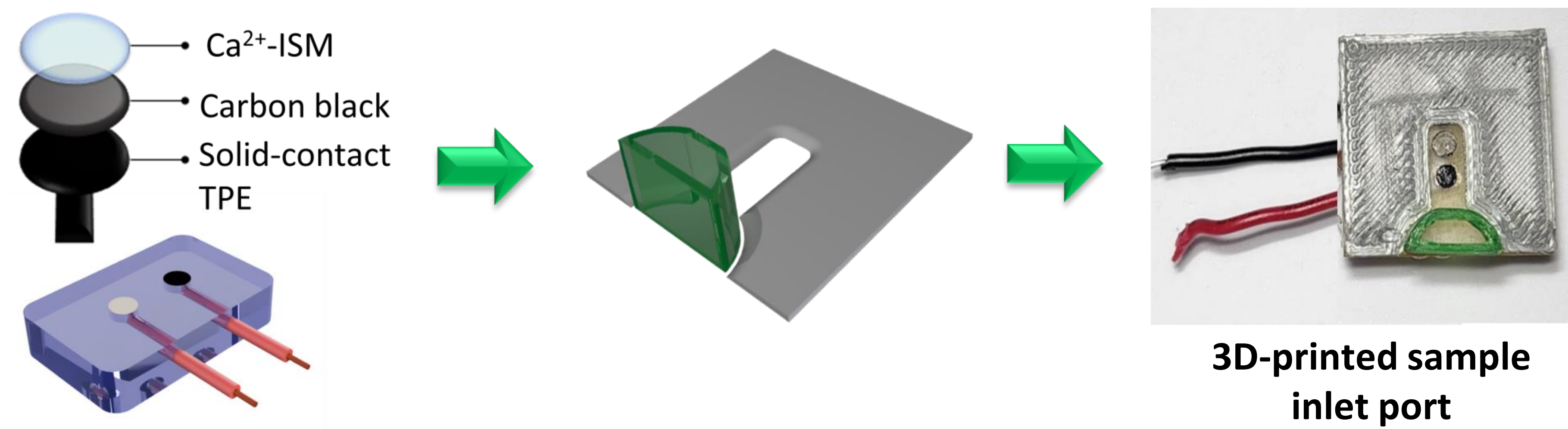
[1] Ozer, T., *Analytical Sciences* 1-11 (2022).

[2] Ozer, T. and Henry, C.S. *Microchimica Acta* **189**(4) (2022).

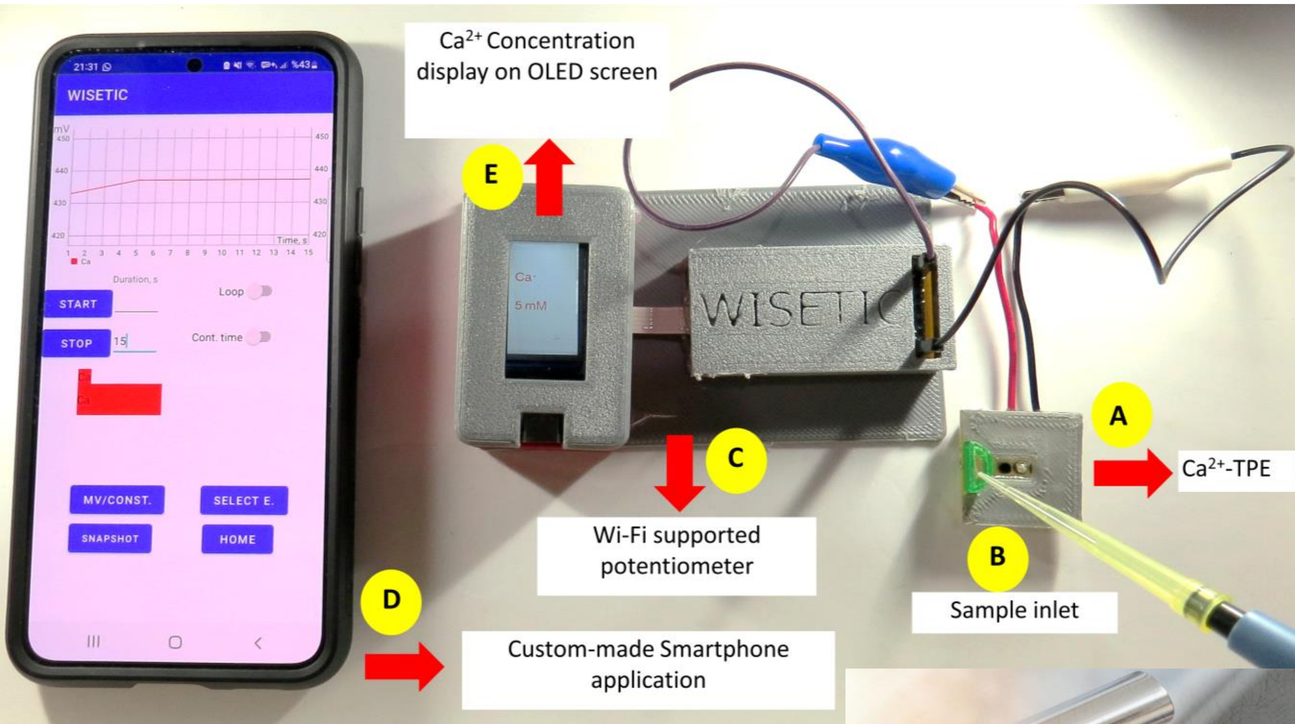
[3] Ozer, T. and Henry, C.S. *Electrochimica Acta*, **404**, 139762 (2022).

Fabrication Process





Detection Process



- **Fast response time (4 s).**
- **A limit of detection of 1×10^{-5} M, 1×10^{-4} M and 1.0×10^{-5} M, respectively for K^+, Na^+ and Ca^{2+}.**
- **Simultaneous detection at the point of need.**

Results

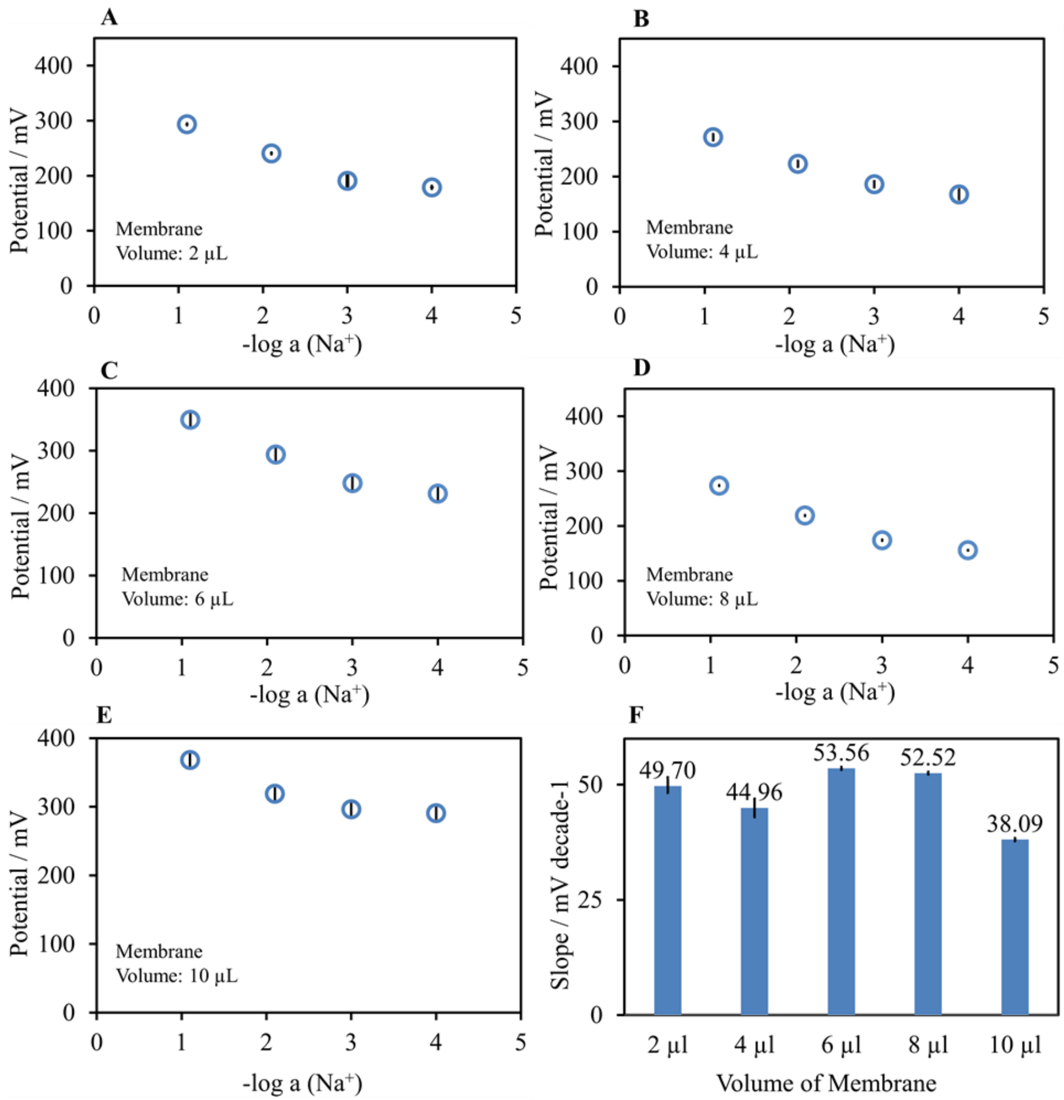


Figure 1. Calibration curves obtained using CB-modified Nano19-based TPEs modified with 2 µL **(A)**, 4 µL **(B)**, 6 µL **(C)**, 8 µL **(D)**, 10 µL **(E)** for ion selective membrane deposition on the TPE surface, measuring 100 µL of a solution containing Na^+ 10^{-4} M, 10^{-3} M, 10^{-2} M, 10^{-1} M. (n=4)

Results

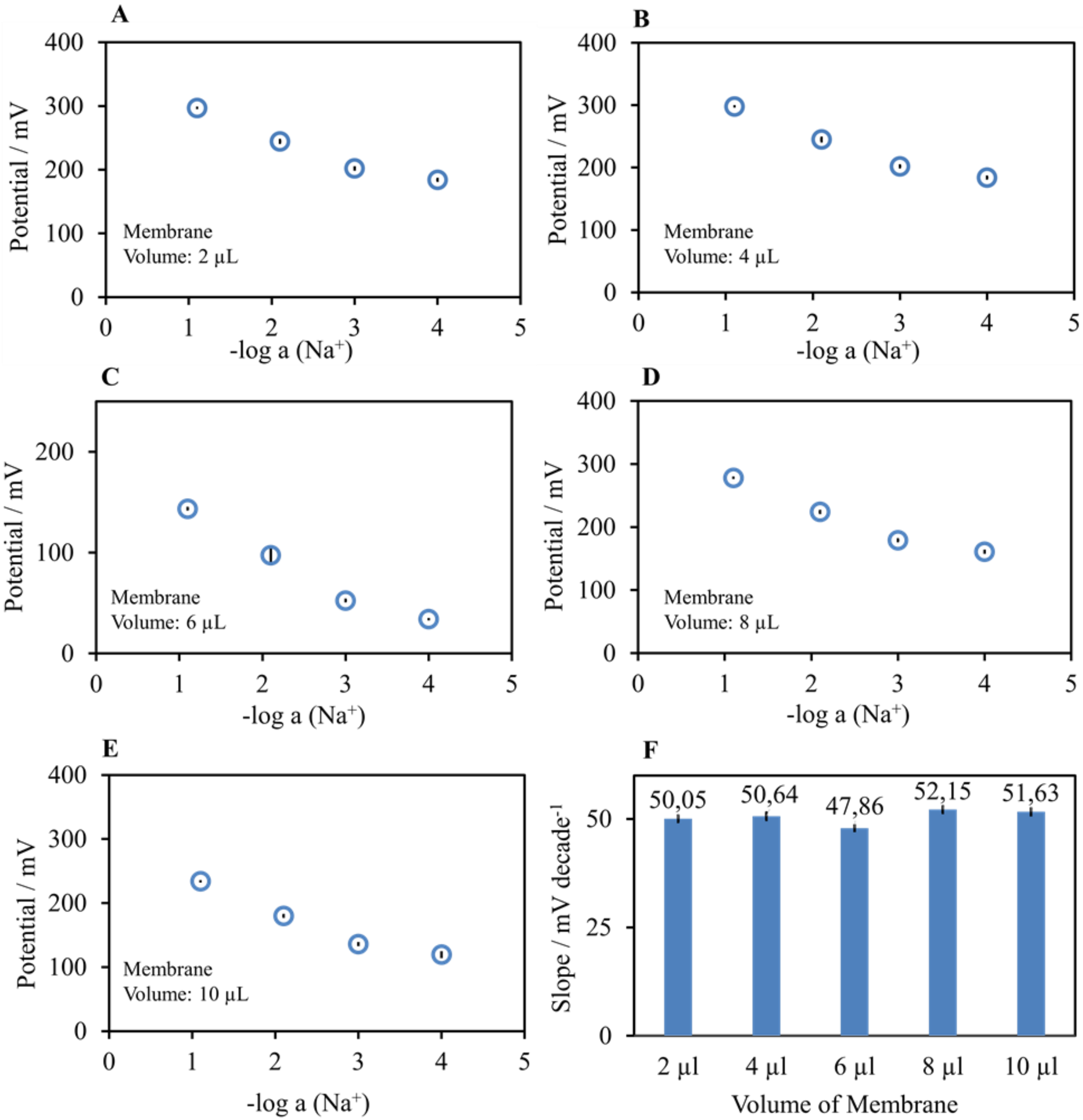


Figure 2. Calibration curves obtained using CB-modified 3805-based TPEs modified with 2 µL **(A)**, 4 µL **(B)**, 6 µL **(C)**, 8 µL **(D)**, and 10 µL **(E)** for ion selective membrane deposition on the TPE surface, measuring 100 µL of a solution containing Na^+ 10^{-4} M, 10^{-3} M, 10^{-2} M, or 10^{-1} M (n=4).

Results

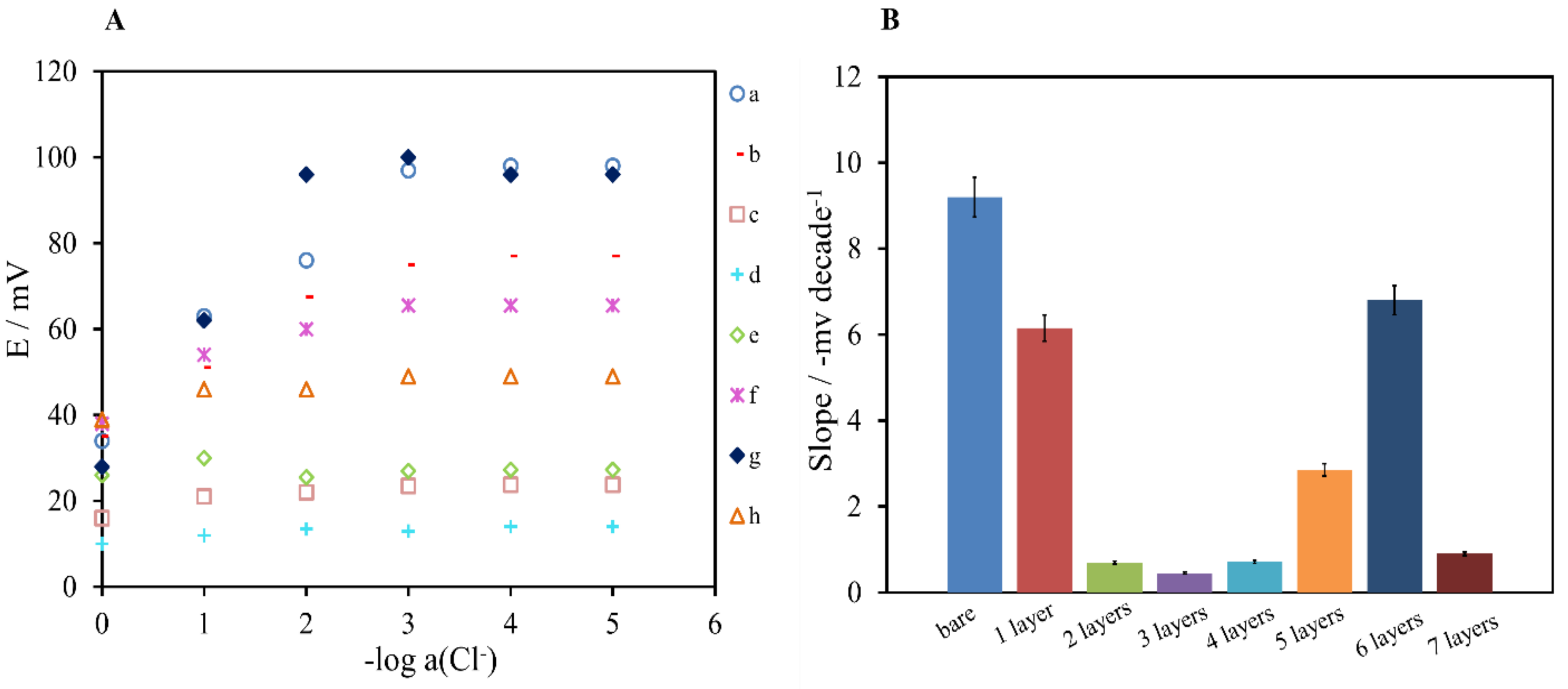


Figure 3. **(A)** Potentiometric responses of the reference TPEs, (a) bare, and (b-h) modified with 2-14 µL (1 layer to 7 layers) of reference membrane solution in varying concentration of Cl^-, 10^{-5}-1 M, in the presence of conditioning step for 16 h in 3 M KCl (n=4). **(B)** Calibration slopes of the reference TPE against variations from 10^{-1} to 10^{-5} M KCl with a 16 h conditioning step (n=4).

Results

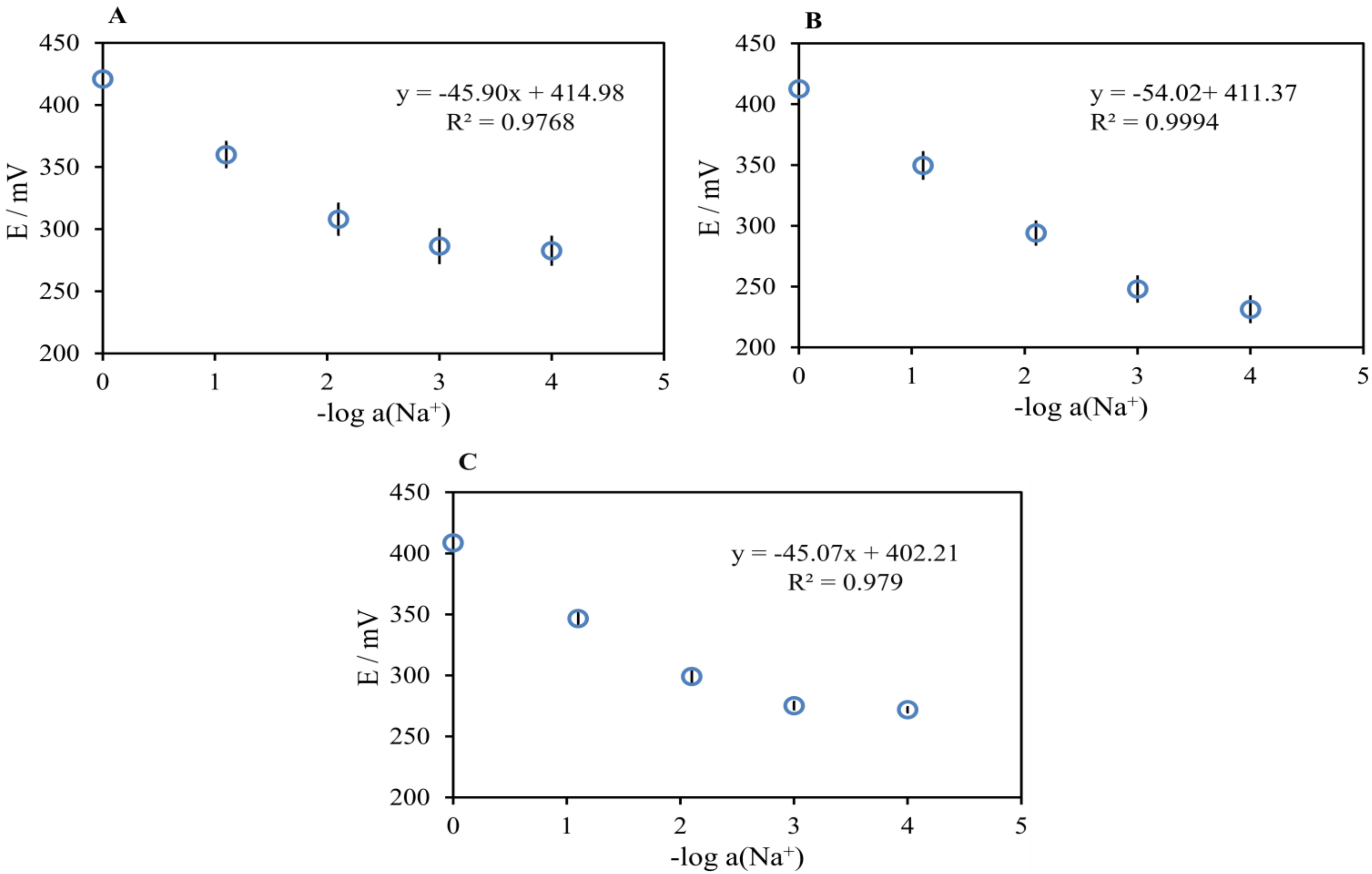


Figure 4. Calibration curves obtained in absence **(A)** and in presence of a 16 h **(B)** and 24 h **(C)** conditioning step in a standard solution of Na^+ 10^{-4} M-1 M. (n=4)

Results

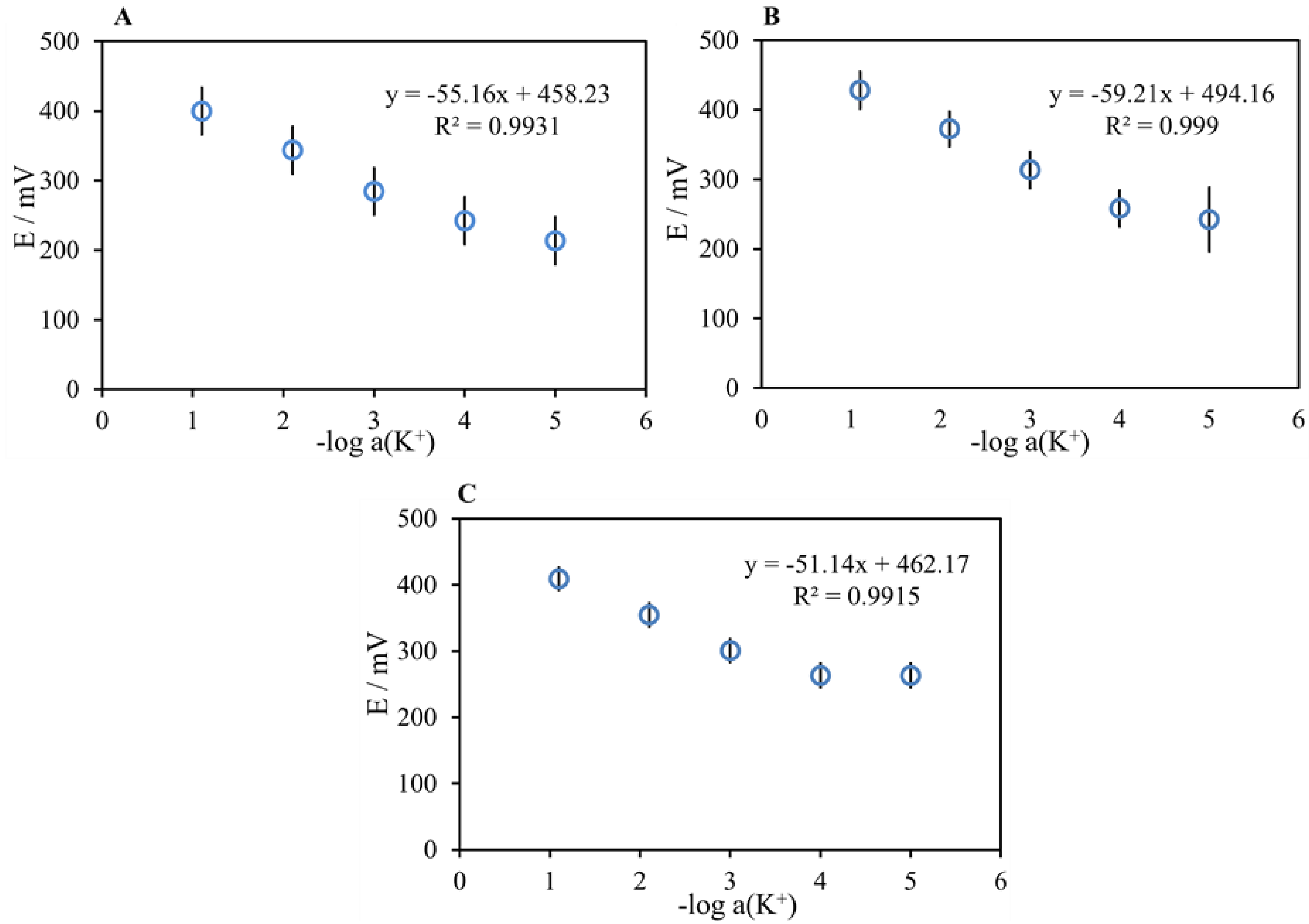


Figure 5. Calibration curves obtained in **(A)** absence and in the presence of a condition step for 16 h **(B)** and 24 h **(C)** in standard solution of K^+ 10^{-5} M-10^{-1} M. (n=4)

Results

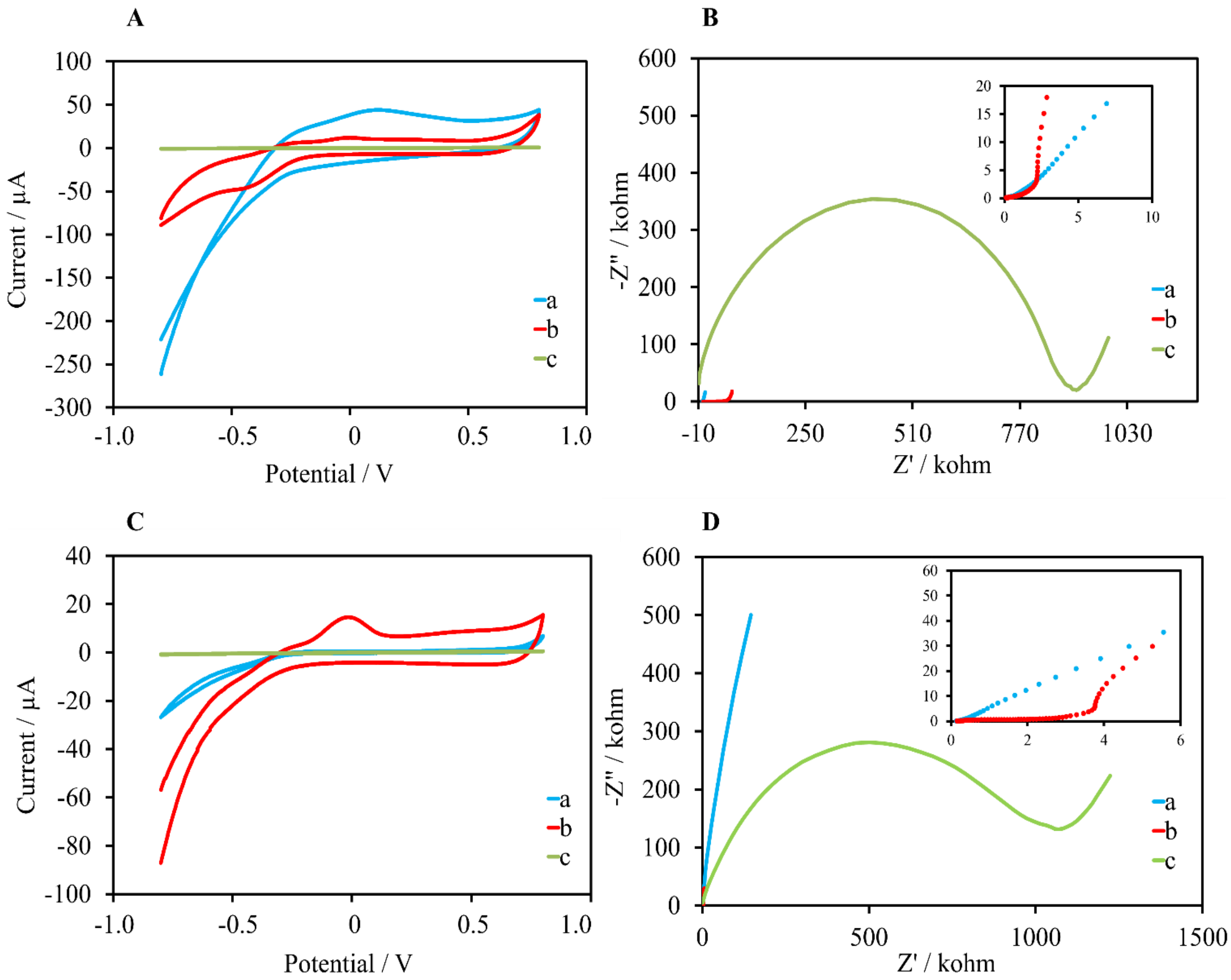


Figure 5.**(A)** Cyclic voltammograms for bare **(a)**, CB-modified **(b)** and Na^+-selective Nano19-based TPE **(c)** in 0.1 M NaCl solution, scan rate: 100 mV/s. (B) Impedance spectra of the Nano19-based TPEs with **(a)** and without CB **(b)**, Na^+-selective-TPE in 0.1 M NaCl solution with a 0.1 Hz−100 kHz frequency range and 10 mV excitation amplitude **(c).** **(C)** Cyclic voltammograms for bare **(a)**, CB-modified **(b)** and Na^+-selective 3805-based TPE **(c)** in 0.1 M NaCl solution, scan rate: 100 mV/s. **(B)** Impedance spectra of the 3805-based TPEs with **(a)** and without CB **(b)**, Na^+-selective-TPE in 0.1 M NaCl solution with a 0.1 Hz−100 kHz frequency range and 10 mV excitation amplitude **(c).**

Results

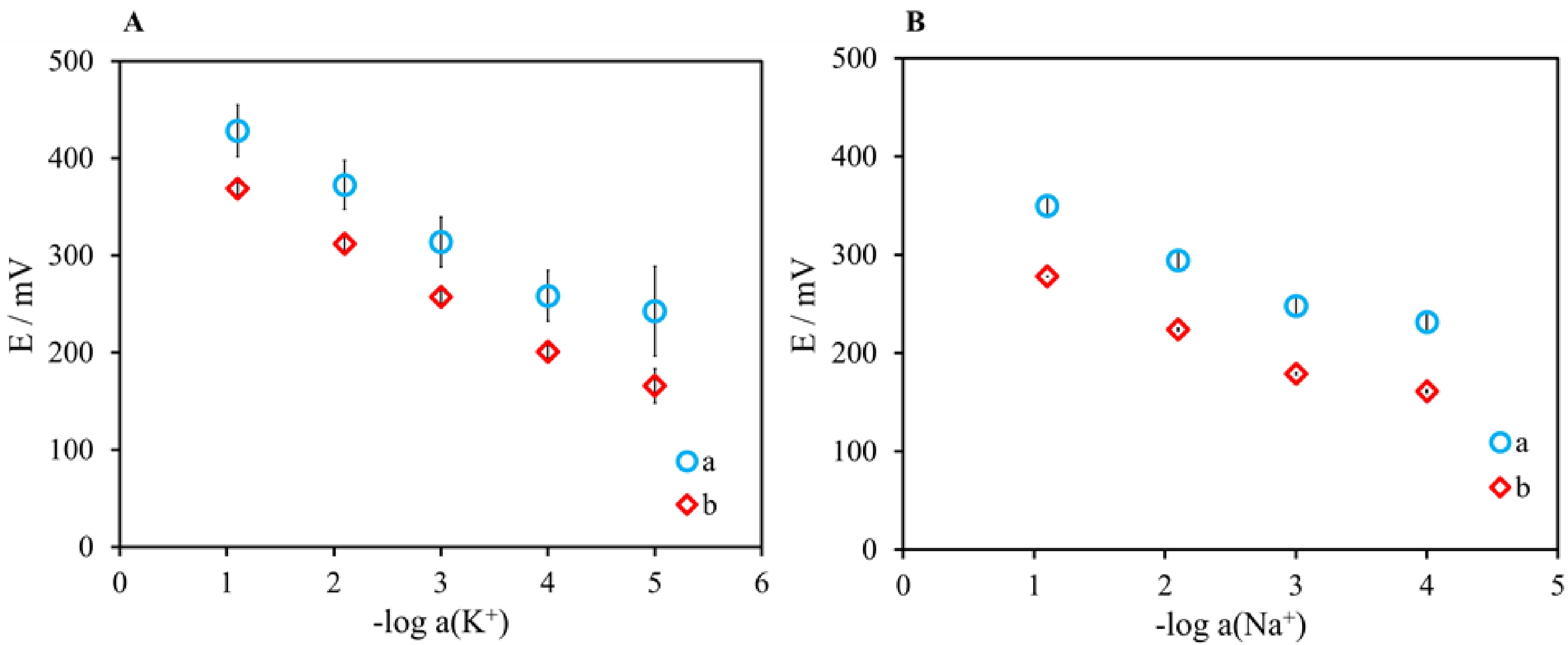


Figure 6. Calibration curve of the **(A)** K^+-selective TPEs fabricated by (a) Nano19 and (b) 3805 as carbon sources for varying potassium activity in water, **(B)** Na^+-selective TPEs fabricated by (a) Nano19 and (b) 3805 as carbon sources for varying sodium activity in water. (n=4)

Results

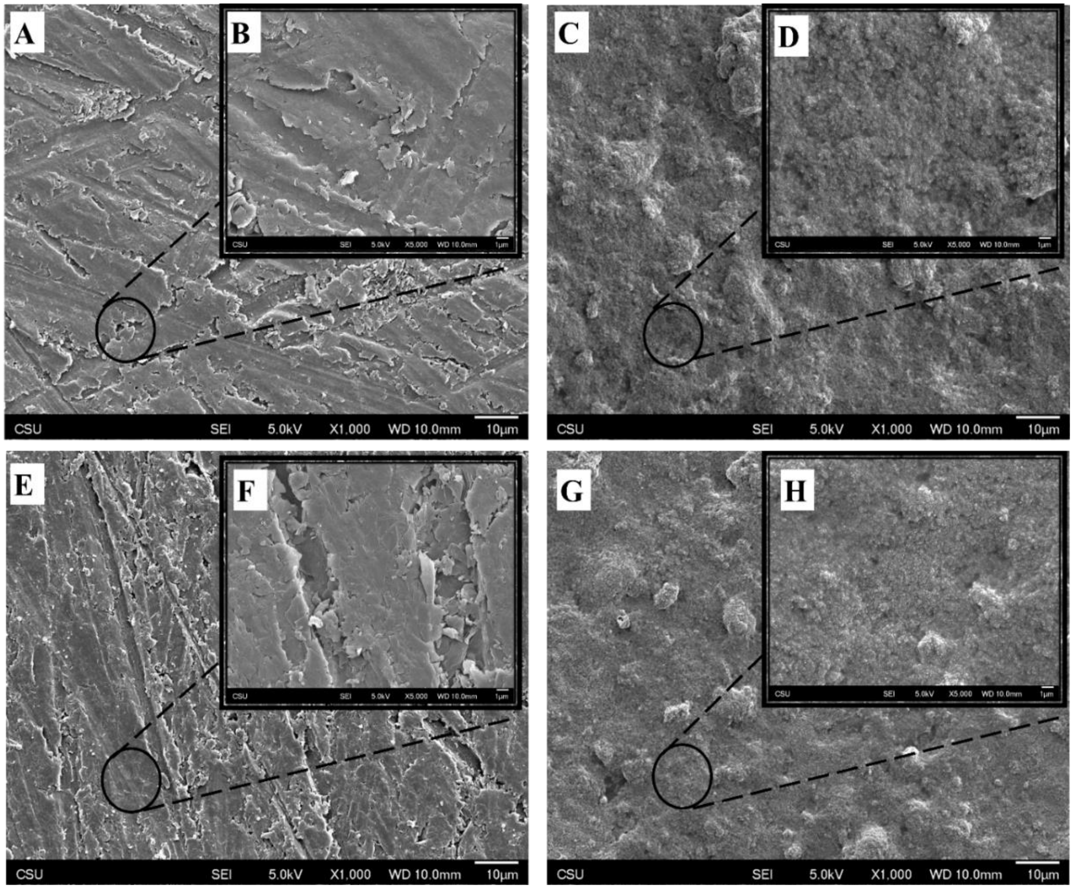


Figure 6.

(A) SEM image and

(B) zoomed-in SEM image of a bare Nano19-based TPE,

(C) SEM image and

(D) zoomed-in SEM image of CB-modified Nano19-based TPE,

(E) SEM image and

(F) Zoomed-in SEM image of a bare 3805-based TPE,

(G) SEM image and

(H) zoomed-in SEM image of CB-modified 3805-based TPE.

Results

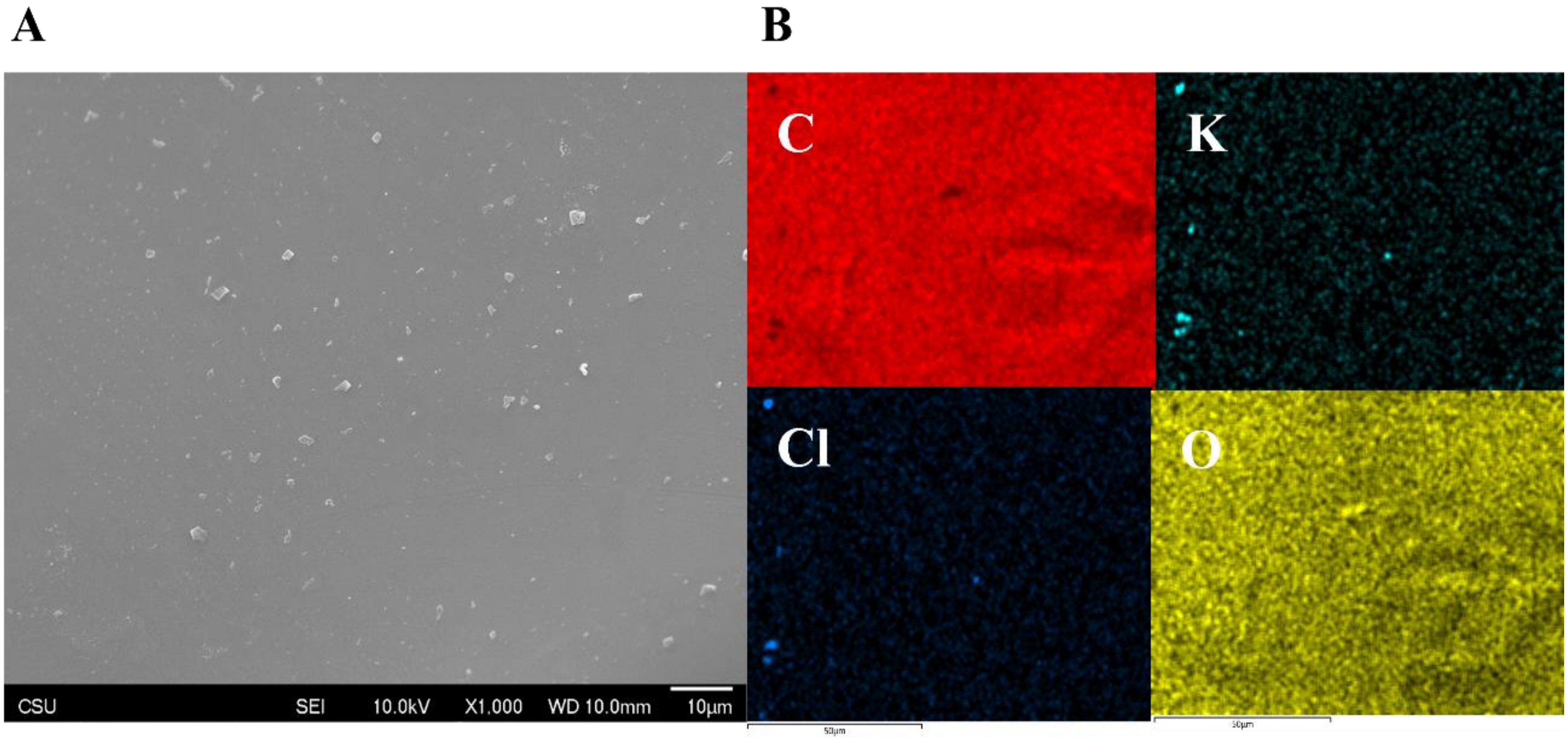


Figure 7.**(A)** SEM image (Scale bar: 10 µm) and **(B)** EDS images of the reference electrode TPE after conditioning in 3 M KCl for 16 h. (Scale bar: 50 µm)

Results

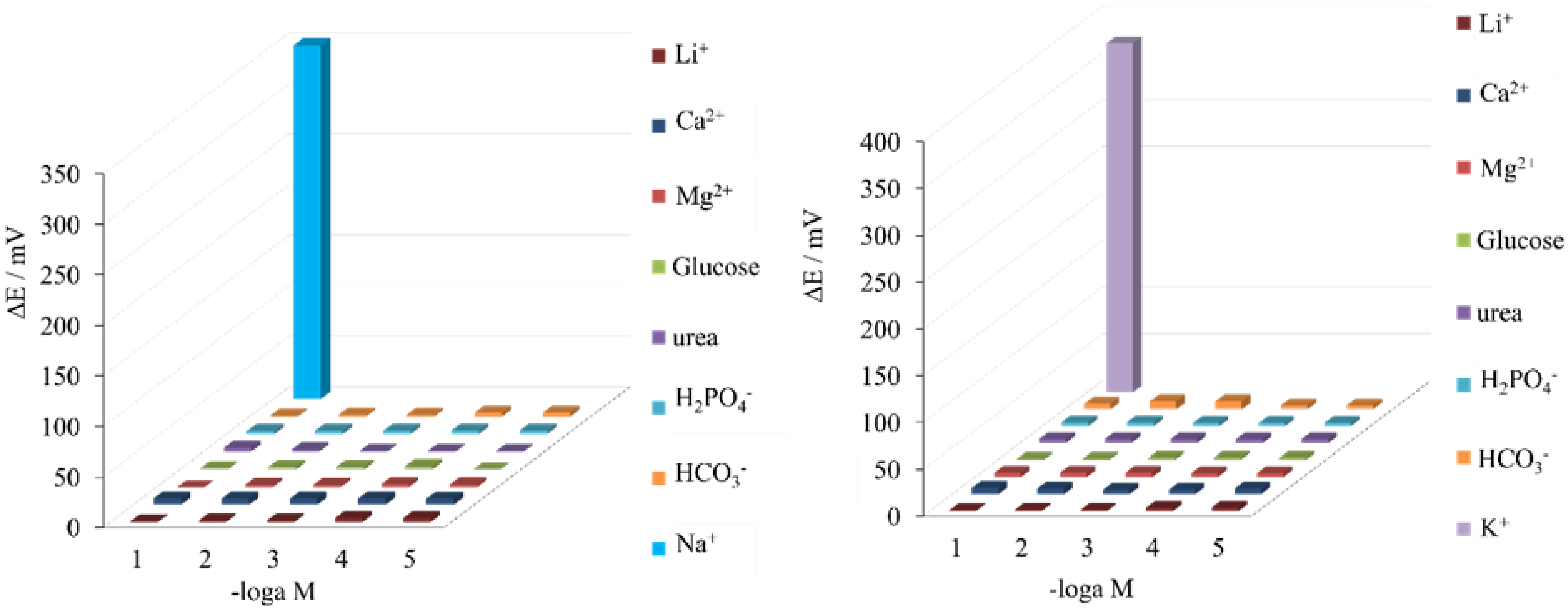


Figure 8. Potentiometric responses (logarithmic activity vs. potential) of **(A)** Na^{+}-selective, **(B)** K^{+}-selective TPEs based on Nano19 towards various interfering metabolites.

Results

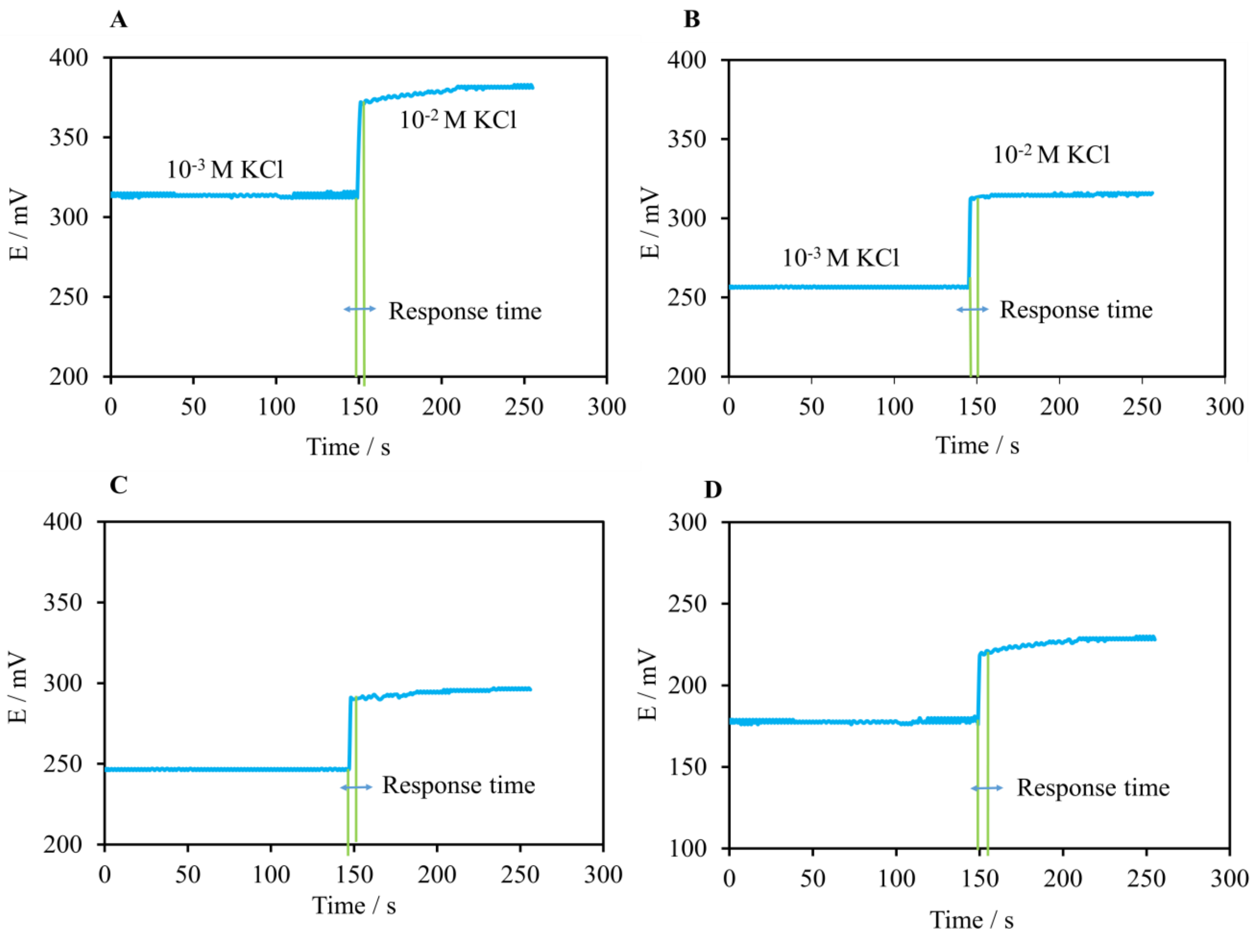


Figure 9. Time dependent potentiometric responses of **(A)** 3805-based, **(B)** Nano19-based K^+-selective TPEs from 10^{-4} M to 10^{-3} M K^+. **(C)** 3805-based, **(D)** Nano19-based K^+-selective TPEs from 10^{-3} M to 10^{-2} M Na^+.

Results

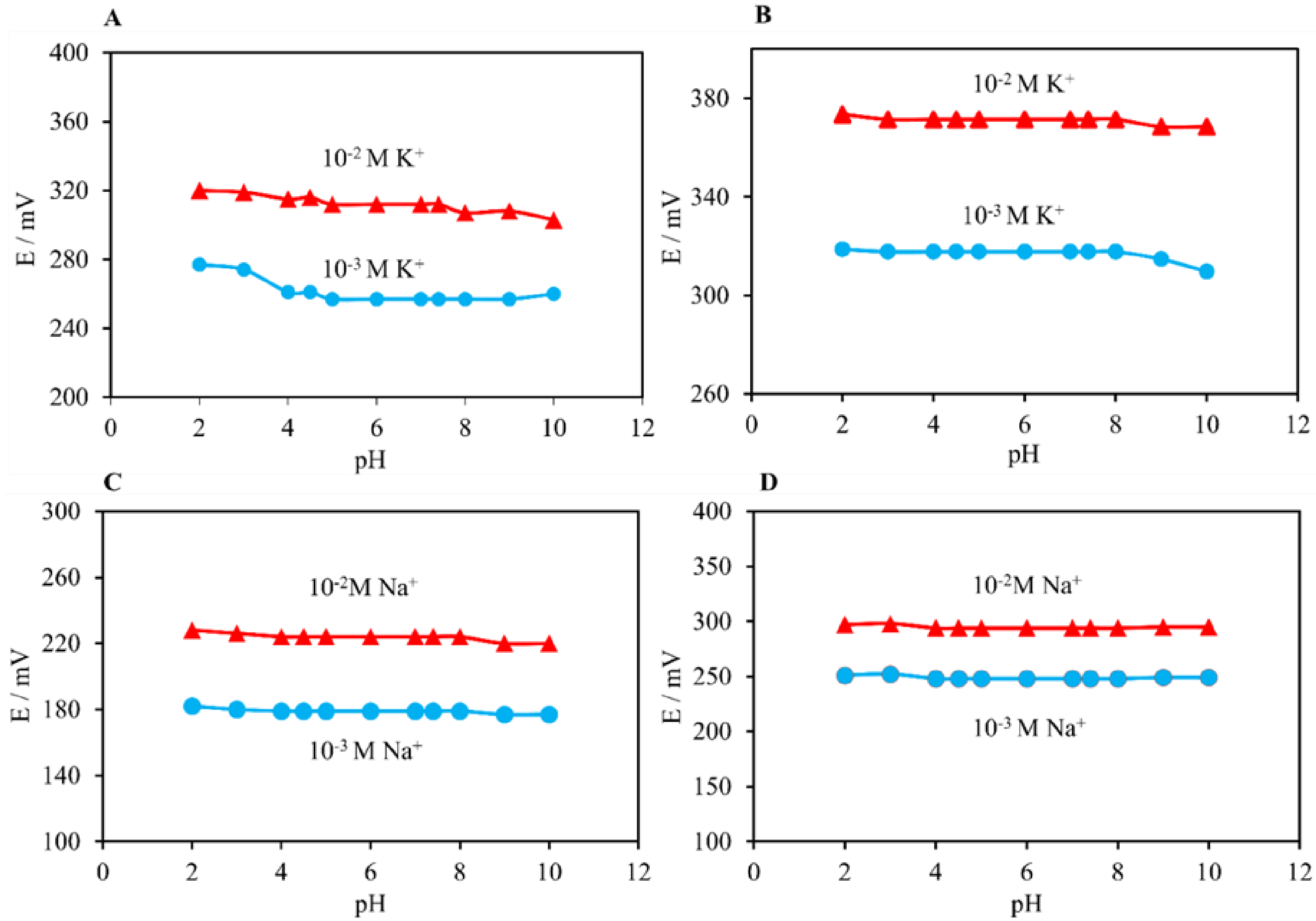


Figure 9. The effect of the pH on the potential response of **(A)** 3805, **(B)** Nano19-based TPEs in 10^{-2} M and 10^{-3} M of K^{+} ion solutions, **(C)** 3805, **(D)** Nano19-based TPEs in 10^{-2} M and 10^{-3} M of Na^{+} ion solutions.

Results

Table 1. Reproducibility and repeatability of Nano19- and 3805-based Na^+-ISEs.

	Slopes (mV/log$_a$ K^+)	Slopes (mV/log$_a$ K^+)	LOD (activity)	Linear Range (activity)
	Reproducibility	Repeatability		
Nano19	53.2, 53.75, 53.73, 53.5, 55.9	55.9, 54.79, 53.17, 52.56, 51.2	10^{-4}	10^{-3} – 1
3805	52.7, 51.6, 52.4, 52.0, 53.17	53.17, 52.69, 54.2, 53.2, 53.14	10^{-4}	10^{-3} – 1

Table 2. Reproducibility and repeatability of Nano19- and 3805-based K^+-ISEs.

	Slopes (mV/log$_a$ K^+)	Slopes (mV/log$_a$ K^+)	LOD (activity)	Linear Range (activity)
	Reproducibility	Repeatability		
Nano19	59.23, 59.01, 59.45, 58.19, 57.99	61.36, 59.23, 59.13, 59.35, 59.84	10^{-5}	10^{-4} – $10^{-1.1}$
3805	58.38, 57.5, 59.27, 58.87, 57.56	57.86, 57.84, 58.39, 58.37, 55.79	10^{-4}	10^{-3} – $10^{-1.1}$

Results

- All nano-sensors were calibrated before the analysis in order to check their correct functioning.
- The ISEs were calibrated using stock primary-ion solutions (10^{-4}- 0.1 M) in a fixed volume of de-ionized water.
- The pH of solutions were adjusted.
- The analysis of water samples was carried out under batch conditions at room temperature. Total analysis time is less than 5 min including calibration process.
- 50 µL of sample was drop-cast on sensor surface and open circuit potential was applied for simultaneous detection of ions to define taste condition of drinking water.


THANK YOU