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Determination of Monoethylene Glycol in Gas Condensate Samples by Microchip Micellar Electrokinetic Chromatography Integrated With Capacitively Coupled Contactless Conductivity Detection

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ABSTRACT

This study describes the use of microchip micellar electrokinetic chromatography (MEKC) integrated with capacitively coupled contactless conductivity detection (C⁴D) for the determination of monoethylene glycol (MEG) in gas condensate samples. The samples were subjected to a liquid–liquid extraction step and then analyzed by chip-based MEKC–C⁴D. For this purpose, sodium dodecyl sulfate (SDS) was used as a surfactant at a concentration of 30 mmol L^{−1} added in 50 mmol L^{−1} phosphate (pH = 9.0). Samples were introduced into microchannels through floating injection mode by applying a voltage of 600 V during 10 s. Separations were performed under an electric field of 82 V cm^{−1} and monitored by C⁴D measurements recorded applying a 1200-kHz frequency sinusoidal wave with 20-V_{pp} excitation voltage. The proposed methodology employing MEKC–C⁴D revealed a linear behavior ($r^2 \geq 0.99$) in the MEG concentration range between 150–450 μmol L^{−1} and LOD equal to 33 μmol L^{−1}. Three gas condensate samples were then analyzed, and the achieved MEG concentration values ranged from 173 to 213 μmol L^{−1}. Recovery experiments provided values between 89 and 102%. Based on the results reported in this study, MEKC–C⁴D devices have demonstrated to be a promising and ecological analytical tool for MEG analysis with huge potential for in-field applications.

1 | Introduction

Monoethylene glycol (MEG) is a diol widely used in the petrochemical industry as it shifts the thermodynamic conditions for hydrate formation out of temperature and pressure ranges, leading to the complete prevention of hydrate formation and blockages in collection pipelines [1]. As well reported in the

literature, MEG is one of the most employed thermodynamic inhibitors due to its properties such as chemical stability, low solubility in nonpolar solvents, reduced toxicity, biodegradability, and the possibility of regeneration through distillation processes [1]. However, the residual presence of MEG in oil, gas, or fuel streams is undesirable and its presence at high concentrations can promote catalyst poisoning, accelerate corrosion processes

Abbreviations: AGREE, Analytical Greenness; MEG, monoethylene glycol.

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in pipelines and storage tanks, and alter the physicochemical properties of automotive fuels [2].

MEG residue is currently determined by redox titration based on an iodometric reaction, which is a time-consuming and a low analytical frequency technique with high waste generation [3, 4]. Instrumental analytical methods like reverse flow injection analysis coupled with spectrophotometric detection [5], gas chromatography coupled with flame ionization detector [6] and liquid chromatography hyphenated with mass spectrometry [7] have been successfully employed for the analysis of MEG. Most of the mentioned methods, especially those based on chromatography, require bulky and expensive instrumentation, that is not always accessible or available only to a few research centers. Besides chromatography-based methods, a few electrophoretic separation modalities including capillary zone electrophoresis (CZE), capillary gel electrophoresis (CGE), and micellar electrokinetic chromatography (MEKC) have demonstrated potential for analyzing different glycols like diethylene glycol (DEG), polyethylene glycols (PEGs), and polypropylene glycols (PPGs) [8]. Among electrophoretic separation modes, MEKC is a suitable strategy for separating neutral compounds due to their partition with micelles. Most of the examples reported in the literature are associated with electrochemical or optical detection and exhibit compatibility with miniaturization [9–11].

In recent years, portable or potentially portable analytical methods have been reported for monitoring MEG content in different samples. Paiva and colleagues explored a glassy carbon electrode modified with electrochemically reduced graphene oxide and gold and palladium nanoparticles for detecting MEG through cyclic voltammetry [12, 13]. The authors reported a LOD of $31 \mu\text{mol L}^{-1}$. Giordano et al. described the development of an alternative analytical method based on microfluidic liquid–liquid extraction followed by electrochemical detection of MEG using nickel disks modified with $\text{Ni}(\text{OH})_2$ nanoparticles [4]. The proposed approach provided LOD equal to 0.65 mmol L^{-1} and satisfactory performance for detecting MEG in gas natural condensate samples. In another study, da Cunha and colleagues developed a point-of-use method based on microemulsification for determining MEG in samples related to natural gas [3]. The authors reported a LOD equal to 54 mmol L^{-1} . More recently, Rezende and colleagues manufactured a portable and 3D-printed batch injection analysis (BIA) cell integrated with cupric oxide sensing electrode for amperometric detection of MEG in gas condensate samples [2]. The portable instrumentation offered LOD equal to 0.45 mmol L^{-1} , short analysis time (ca. 30 s), and low sample volume requirement ($15 \mu\text{L}$).

Chip-based electrophoresis devices integrated with capacitively coupled contactless conductivity detection (C^4D) have emerged as powerful and portable platforms for analytical applications involving different electromigration methods [14–19]. Recent examples of applications including the separation and detection of post-blast explosive residues [20], the determination of naphthenic acids in produced water [21], screening of seized cocaine samples [22], food authenticity [23], the detection of galantamine in human plasma [24] and analysis of soil nutrients [25] have been successfully reported in the literature. Although all the mentioned examples are associated with the use of free-solution

electrophoresis mode, MEKC has also been implemented in chip-based format for analyzing several classes of analytes [9, 10, 26, 27]. However, to the best of our knowledge, the analysis of MEG using this separation mode coupled with C^4D has not been reported yet.

In this way, this study reports for the first time the development of a methodology based on MEKC for analyzing MEG in gas condensate samples using commercial chip-based electrophoresis devices integrated with C^4D . MEG was initially extracted from oil samples by single-phase liquid–liquid extraction approach and then analyzed by MEKC exploiting previously optimized experimental conditions. The feasibility of the developed approach was then demonstrated by analyzing MEG in three gas condensate samples.

2 | Experimental Section

2.1 | Chemicals and Materials

Sodium hydroxide, sodium dodecyl sulfate (SDS), sodium dibasic phosphate, and MEG were purchased from Sigma Aldrich (St. Louis, MO, USA) and used as received. Stock solutions were prepared weekly using ultrapure water ($18.2 \text{ M}\Omega \text{ cm}$) processed through a purification system (Direct-Q 3, Millipore, Darmstadt, Germany). Before the analysis, sample and stock solutions were filtered through nylon filters with a $0.22 \mu\text{m}$ pore diameter. Subsequent dilutions were carried out using ultrapure water. All experiments were performed at room temperature ($23 \pm 1^\circ\text{C}$).

2.2 | Instrumentation

MEKC experiments were performed using an eDAQ system (Denistone East, NSW, Australia) (Figure S1A), composed of an ET225 microfluidic platform (Figure S1B), a high voltage sequencer (HVS) Model ER230 and a C^4D model ER455 (Figure S1C). The injection and separation voltages were applied by the HVS and controlled through the software Sequencer.

MEKC-based separations were performed on glass microchips model ET190 supplied by Micronit Microfluidics B.V. (Enschede, Netherlands) (see Figure S1D). The microchip is designed in a double-T format (gap of $100 \mu\text{m}$) containing injection and separation channels defined with width and depth equal to $100 \mu\text{m}$ and $10 \mu\text{m}$, respectively. The total and effective separation channel lengths were 85 and 77 mm, respectively. For C^4D measurements, the detection cell was composed of four sensing electrodes of platinum with a tantalum adhesion layer. The distance between excitation and detection electrode was $250 \mu\text{m}$.

The sample conductivity was measured using a microprocessor-based benchtop conductivity meter (model W12D, Bel Engineering). For this purpose, standard solutions of MEG were prepared at different concentrations ($100\text{--}550 \mu\text{mol L}^{-1}$) in 50 mmol L^{-1} sodium phosphate (pH 9.0) in the absence and presence of 30 mmol L^{-1} SDS.

2.3 | Electrophoresis and C⁴D Procedures

Electrophoresis microchannels were electrokinetically conditioned with 0.1 mol L⁻¹ NaOH through the application of 800 V for 10 min followed by a rinsing step with ultrapure water for an additional 10 min. Later, channels were electrokinetically conditioned with the background electrolyte (BGE) composed of 50 mmol L⁻¹ sodium phosphate dibasic solution containing 30 mmol L⁻¹ SDS by applying the voltages selected for electrophoretic separations. The sample was electrokinetically injected by floating mode. For this purpose, a voltage of 600 V was applied to the sample reservoir keeping the sample waste grounded during 10 s to ensure channel filling with sample. Afterward, a separation voltage of 700 V was applied to the buffer reservoir, keeping the buffer waste reservoir grounded.

C⁴D measurements were recorded applying a 1200-kHz frequency and an excitation voltage of 20 V_{peak-to-peak}. PowerChrome (eDAQ, Denistone East, NSW, Australia) software was used for data acquisition. All experiments were performed at 23 ± 1°C.

2.4 | Samples

Three natural gas condensate samples supplied by the company Petrobras were analyzed. Initially, MEG present in these samples was extracted through a liquid–liquid extraction method using BGE as aqueous phase. The extraction was carried out in a single step by vortex mixing for 3 min of the microtube containing 200 µL of sample and 200 µL of BGE, followed by a 15-min rest until complete phase separation. The aqueous phase was analyzed without any other treatment.

2.5 | Green Analytical Chemistry Metrics for MEG Analysis

To evaluate the environmental sustainability profile (greenness) of the proposed MEKC-MSE method, in comparison with conventional HPLC-MS and GC-MS methods for MEG analysis, the free AGREE software available at <https://mostwiedzy.pl/AGREE> (version 0.5, 2020) was used [28].

3 | Results and Discussion

3.1 | Optimization of Experimental Parameters

In CZE mode, all neutral analytes have zero effective mobility and, thus, migrate at the same velocity as the EOF. Therefore, MEKC has been selected as ideal separation mode for MEG analysis. In MEKC separation mode, a surfactant is added to the BGE allowing it to act as a pseudo-stationary phase and, thus, enabling the separation of neutral compounds based on partition between analyte and micelles. SDS, an anionic surfactant, was used to form micelles in a 50 mmol L⁻¹ phosphate (pH 9.0) BGE [29]. In this BGE, MEG migrates with a mobility that is a fraction of that of micelles toward the cathode under normal polarity [30].

Figure 1 displays typical electropherograms showing the detection of MEG with C⁴D.

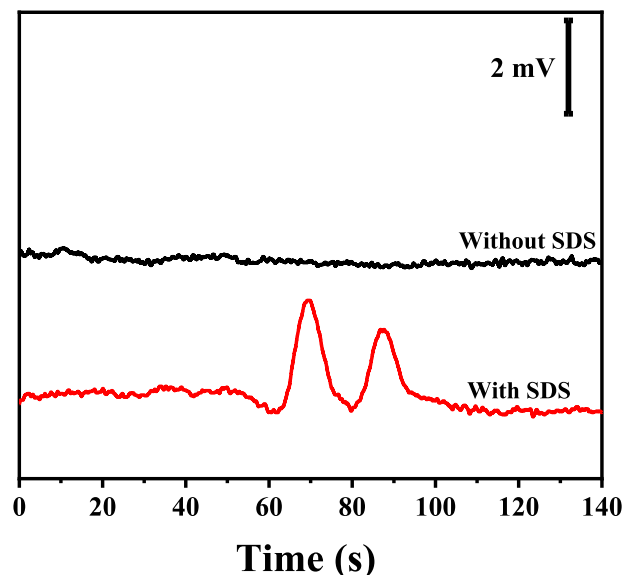


FIGURE 1 | Electropherograms showing the detection of MEG prepared in BGE composed of 50 mmol L⁻¹ phosphate (pH 9) with and without SDS (30 mmol L⁻¹). Floating injection: 600 V/10 s. Separation voltage: 700 V. Detection operational parameters: 1200-kHz frequency and 20 V_{peak-to-peak} amplitude.

Since the electropherograms showed more than one peak, even when standard MEG solutions were diluted in the BGE, a spiking test was performed with standard solutions of MEG prepared in three concentration levels (300, 500, and 700 µmol L⁻¹). As can be seen in Figure 2A, the addition of standard solutions of MEG promoted an increase on the peak detected at ca. 85 s. Thus, the peak detected at ~ 65 s is a system peak.

The mechanism of conductometric detection in MEKC is complex and has received limited investigation since its introduction [31]. It can, however, be rationalized by three levels of phenomena.

First, the effect of neutral analyte on electrolyte conductivity must be considered. Although no direct contribution is expected from an uncharged species, conductivity may change through modification of the molar ionic conductance of another species (e.g., complexation or adduct formation). Additionally, the analyte may alter the medium viscosity, particularly at high concentrations. The constant conductivity observed in Figure 2B for the BGE without SDS indicates that such effects are negligible in the range studied.

In Figure 2B, it can also be observed that the addition of SDS makes the BGE conductivity dependent on the concentration of MEG, and therefore, the effect must be attributed to this analyte acting on micellar mobility. Indeed, it is expected that a species such as MEG, being an alcohol, modifies micelle size and composition. Once again, the behavior is not straightforward, since on the one hand, incorporation of MEG molecules increases micelle size which should lead to reduced mobility whereas, on the other hand, enlargement of the micellar aggregate lowers the density of negative charges from dodecyl sulfate, favoring dissociation of Na⁺ ions retained on the micelle surface which should contribute to increased mobility. In the present case, we

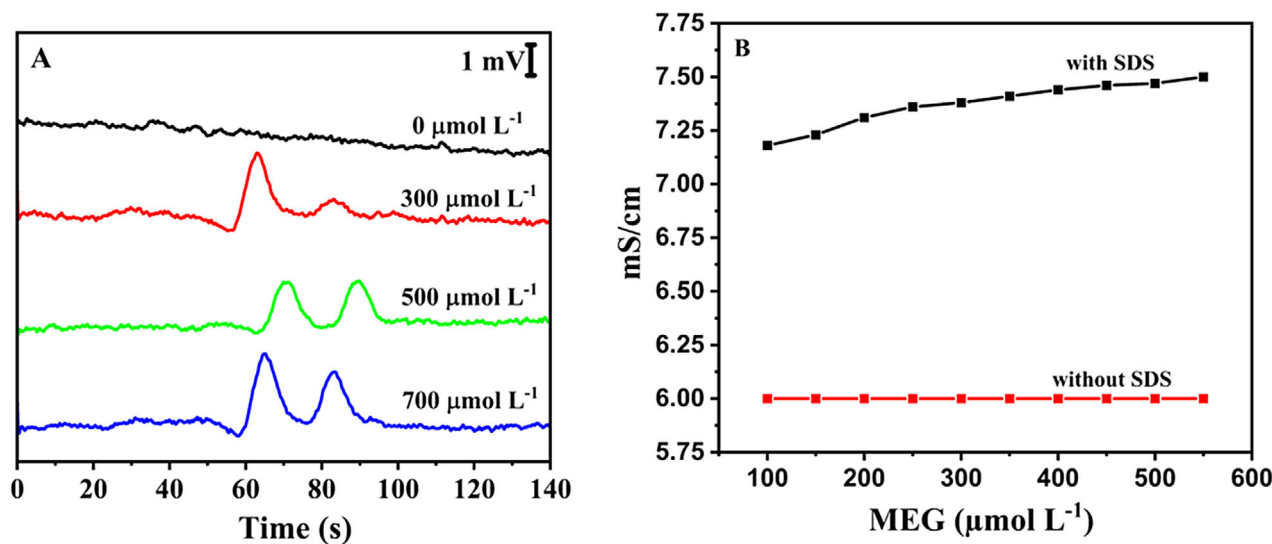


FIGURE 2 | (A) Electropherograms showing the detection of MEG at different concentrations (0, 300, 500, and 700 $\mu\text{mol L}^{-1}$). (B) Conductivity measurements for MEG solutions prepared in BGE with and without SDS. Other experimental and instrumental conditions: see Figure 1.

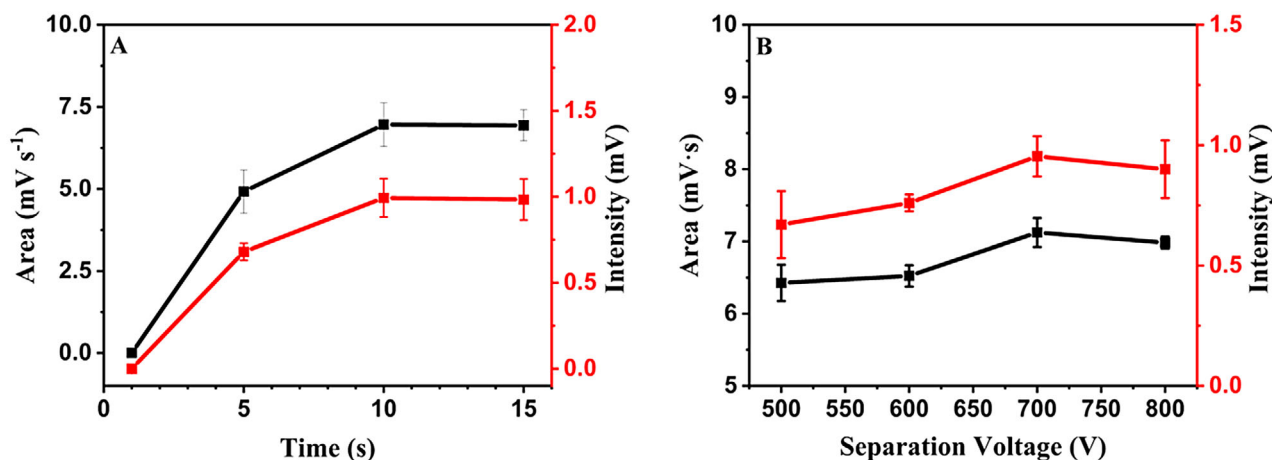


FIGURE 3 | Effect of the (A) injection time and (B) separation voltage on MEG (300 $\mu\text{mol L}^{-1}$) peak area and intensity. In (A), the voltage values applied for injection and separations were 600 and 700 V, respectively. In (B), the injection was performed applying 600 V during 10 s. Other experimental and instrumental conditions: see Figure 1.

observe that the overall effect is an increase in mobility, because the conductivity of the SDS-containing BGE increases, as shown in Figure 2B.

Finally, it should also be considered that these phenomena occur during an electrophoretic migration process, which takes place simultaneously with longitudinal diffusion. Thus, even the kinetics of micelle formation may become significant, potentially explaining the appearance of system peaks, such as that observed in Figure 2A.

The instrumental conditions involving the injection time as well as the voltages applied for sample injection and separation were also optimized. Initially, the voltage applied for injection was evaluated keeping constant the separation voltage (700 V) and the injection time (10 s). Then, a standard solution of 300 $\mu\text{mol L}^{-1}$ MEG was injected into microchannel and

electropherograms were recorded varying the injection voltage between 300 and 700 V. The peaks with higher intensities were observed when sample was introduced into microchannels applying 600 V (Figure S2). Therefore, this voltage was selected as optimal and kept constant for the subsequent experiments.

Regarding the injection time, the same MEG solution was introduced into microchannels varying the floating time between 1 and 15 s and keeping constant the injection and separation voltages at 600 and 700 V, respectively. As can be seen in Figure 3A, the peak related to MEG was only observed when solution was injected under floating time longer than 5 s. In addition, it was observed that increasing the injection time provided an increase on the MEG peak intensity. It was somewhat expected once the floating time impacts in the full loading channel intersection and, consequently, the injection channel. For floating times longer

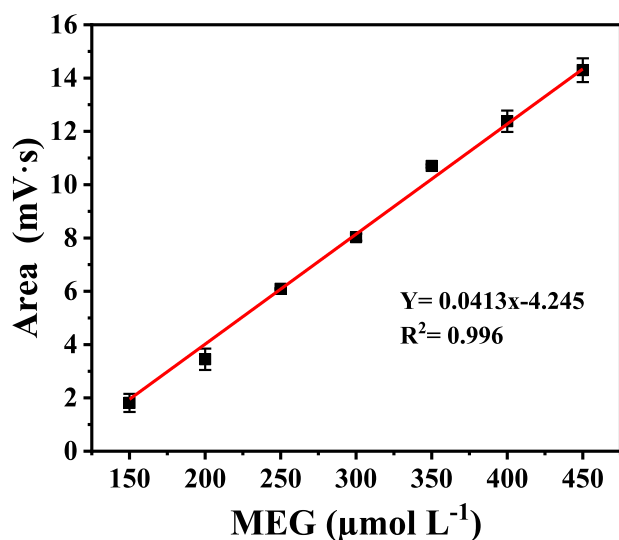


FIGURE 4 | Analytical curve showing the effect of the MEG concentration on the recorded peak area. Experimental and instrumental conditions: see Figure 1.

than 10 s, no significant difference was observed for the recorded MEG peak, thus indicating the complete filling of channel with sample solution. The floating time of 10 s was selected as ideal injection time and kept constant for the subsequent experiments.

Lastly, the separation voltage was also investigated by keeping the injection voltage (600 V) and floating time (10 s), as previously defined. Electropherograms were recorded under the application of high voltage values between 500 and 800 V. As can be seen in Figure 3B, the use 700 V for the separation provided peaks with higher intensity and area, better peak symmetry and greater baseline stability. Furthermore, under these conditions, the system peak does not interfere with the signal of the analyte under study, which was observed when using higher potentials.

3.2 | Analytical Performance

3.2.1 | Analyses of Gas Condensate Samples

The analytical performance of the proposed methodology was investigated to evaluate its feasibility for analyzing the MEG concentration levels in natural gas condensate samples. As shown in Figure 4, the use of chip-based MEKC integrated with C⁴D provided a linear behavior ($r^2 = 0.996$) for MEG in the concentration range between 150 and 450 $\mu\text{mol L}^{-1}$ (Figure S3). The limits of detection (LOD) and quantification (LOQ) were calculated and presented values equal to 33 and 109 $\mu\text{mol L}^{-1}$, respectively. The LOD and LOQ values were achieved based on the signal-to-noise ratios of 3 and 10, respectively.

The repeatability was assessed for 12 consecutive injections of MEG solution previously prepared at concentration of 200 $\mu\text{mol L}^{-1}$. The RSD values obtained for migration time and peak area were 4.8% and 4.2% respectively. In addition to migration time and peak area, the separation efficiency was calculated and presented values equal to $(7.8 \pm 0.4) \times 10^3$ plates m^{-1} .

The analytical merit figures achieved using on-chip MEKC-C⁴D were compared to other techniques found in the literature for MEG analysis. As can be seen in Table 1, although the proposed approach has offered the narrowest concentration linear range, it provided lower LOD and LOQ values when compared to chromatographic [6, 7] and spectrophotometric [5] methods. In addition, the detectability levels achieved were within the same magnitude range when compared to electrochemical approaches that employed modified working electrodes [12, 13]. Considering the comparison presented in Table 1, it is important to highlight that conventional techniques like chromatography and spectrophotometry require expensive and bulky instrumentation as well as larger amount of sample and reagents. In addition, they involve time-consuming procedures associated with sample pretreatment steps [6, 7]. Iodometric titration, employed as the reference method by Petrobras for MEG determination in regeneration samples, also offers low-cost implementation and simple instrumentation, but requires larger sample volumes, is time-consuming and laborious, and involves extensive use of reagents. In addition, its analytical performance, although adequate for routine analysis [3], does not reach the lower detection limits achieved by the proposed method.

As summarized in Table 1, the MEKC-C⁴D device provided additional advantages when compared to other reports found in the literature including reduced consumption of sample and reagents, short analysis time, minimal sample treatment requirements, and low waste generation.

3.2.2 | Analyses of Gas Condensate Samples

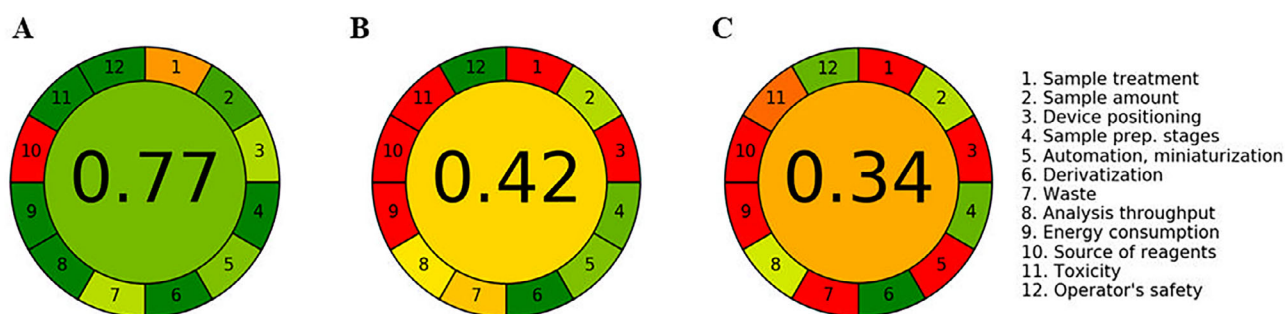
The developed methodology was used to quantify MEG in three gas condensate samples received from Petrobras. The analyses were performed with all optimized parameters kept constant. Considering the information extracted from the analytical curve, the MEG concentrations were calculated, and the values found for samples 1, 2, and 3 were 173 ± 7 , 213 ± 18 , and 183 ± 8 $\mu\text{mol L}^{-1}$, respectively. The accuracy of the developed method was studied through spiking the diluted petrochemical samples with MEG standard solutions prepared at two concentration levels (100 and 150 $\mu\text{mol L}^{-1}$). The recoveries calculated for all samples varied from 89 ± 3 to $102 \pm 5\%$, thus demonstrating satisfactory performance for quantifying MEG in complex samples.

3.3 | Greenness

After evaluating the 12 principles of green analytical chemistry using the AGREE software, the overall values obtained were 0.77 for chip-based MEKC devices, 0.42 for GC-MS and 0.34 for LC-MS, as displayed in the pictograms in Figure 5. The high score of the proposed methodology reflects its strong adherence to the GAC principles, driven mainly by miniaturization and the possibility of automation, which contributes to a significant reduction in sample amount (item 2), energy consumption (item 9), and waste generation (item 7). Furthermore, the reduced use or absence of toxic and hazardous reagents (items 10 and 11) reinforces its environmentally safer profile. In contrast, classical

TABLE 1 | Comparison of techniques and figures of merit for MEG analysis with other methods found in the literature.

Analytical technique	Sample volume	Linear range (mmol L ⁻¹)	LOD (mmol L ⁻¹)	LOQ (mmol L ⁻¹)	Ref.
Microemulsification-based method (MEC)	600 µL	NR	54	NR	[3]
Electrochemistry	NR	2–12	0.651	NR	[4]
LC-MS/MS	40 µL	1.6–64	0.563	4.83	[7]
GC-FID	1 µL	1.6–81	0.403	0.806	[6]
Spectrophotometry	5.0 mL	0.03–1.61	0.161	NR	[5]
Electrochemistry	NR	0.24–1.43	0.049	0.162	[12]
Batch injection analysis with electrochemical detection	15 µL	1–25	0.45	NR	[2]
Electrochemistry	NR	0.09–0.18	0.0315	0.104	[13]
Iodometric titration	50–100 mL	NR	NR	NR	[4]
MEKC-C ⁴ D	200 µL ^a 300 pL ^b	0.15–0.45	0.0210	0.635	This study

^aSample volume used in the extraction procedure.^bSample volume introduced inside separation channel floating injection mode; NR = non reported.**FIGURE 5** | Representation of the values obtained by the AGREE metric for different analytical techniques: micellar electrokinetic chromatography (A), gas chromatography coupled to mass spectrometry (B), and liquid chromatography coupled to mass spectrometry (C).

analytical methods such as HPLC and GC showed inferior metrics due to the use of organic solvents, energy involved in the process and lower operational safety. Therefore, MEKC devices not only prove to be technically effective but also stand out as a more sustainable, safer approach aligned with modern GAC practices.

4 | Concluding Remarks

In this report, we have demonstrated for the first time the feasibility of chip-based MEKC-C⁴D devices for determining MEG analysis in petrochemical matrices. The developed analytical methodology provided satisfactory analytical performance including efficient extraction procedure (< 20 min) using reduced amount of sample (200 µL), rapid electrophoretic analysis (<100 s), reduced sample injection volume requirement (~300 pL), minimal waste generation, and low detectability levels (33 µmol L⁻¹). When compared to conventional chromatography-based analytical techniques or reference method involving redox titration, the use of chip-based MEKC-C⁴D devices provided better greenness metrics mainly due to inherent advantages like miniaturization, low sample and reagent requirements, low energy consumption, simpler

and potentially portable instrumentation. Based on the achievements reported in this study, MEKC-C⁴D devices emerge as powerful analytical tools for offshore applications, opening a new gate for investigating neutral compounds in petrochemical samples.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.

Supporting File: elps70070-sup-0001-SuppMat.pdf.