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DEGRADATION STUDY OF DEEP EUTECTIC SOLVENTS IN CO_2 CAPTURE TECHNOLOGIES

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CO_2 from post-combustion flue gases can be captured using chemical solvents, the main one MEA30% aqueous, or in newer, greener solvents, namely deep eutectic solvents. In this study, the three solvent's chemical degradation was tested: MEA 30%, ChCl:EG 1:2 and ChCl:MEA 1:8, after a corrosion test on a stainless-steel plate. As expected, the amine dissolved in water had corroded the metallic sample and the solvent suffered oxidation reactions, on the other hand the DES samples protected the plate, and their composition remained stable. The chemical and thermal stability is one of the main advantages of the DES, proven also in the present test.

Keywords: deep eutectic solvents, solvent degradation, FTIR, CO_2 capture, XRF

1. Introduction

In November 2021, researchers at the GCP (Global Carbon Project) estimated that global CO_2 emissions from energy and industry raised by 4.8% in 2021, after a 5.8% decline in 2020 (almost 2 Gt CO_2) [1] [2], due to the pandemic period. Despite the decline in 2020, global energy-related CO_2 emissions remained at 31.5 Gt, which contributed to CO_2 reaching its highest ever average annual concentration in the atmosphere of 412.5 parts per million in 2020 – around 50% higher than when the industrial revolution began. China, India, and the European Union all had significant increases, suggesting that any climate effect from the pandemic was fleeting.

In 2021 global energy-related CO_2 emissions were projected to grow by 4.8% as demand for coal, oil, and gas rebounded with the economy. The increase of 1500 Mt CO_2 was the most dramatic single increase since the carbon-intensive economic recovery from the global financial crisis more than a decade ago; the global emissions in 2021 were around 400 Mt CO_2, or 1.2%, below the 2019 peak.

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2. CO_2 capture using DES

CO_2 can be captured in different types of solvent using post-combustion methods [3-7]. One solvent type is represented by the deep eutectic solvents DES, analogues to ionic liquids IL. They are derived from salts as the hydrogen bond acceptor HBA (e.g., choline chloride) and at least one or more hydrogen bond donors HBD, such as amides, amines, alcohols, and carboxylic acids [8, 9]. Deep eutectic solvents present several advantages: no chemical reaction with water, low volatility, high thermo-stability, lower price compared to IL, nontoxic, easy preparation, lower melting point than the mixture's constituents and environmentally friendly.

3. DES degradation

Oxidative degradation is caused by the oxygen present in the gas stream [10]. This occurs mainly in the absorption column, between 40-70°C, in the rich solvent at the outlet of the heat exchanger, and in the desorption column, where temperatures are between 100-145°C. This type of degradation has become a main issue while separating CO_2 from flue gases, where the O_2 concentration usually varies between 3 and 5%. The electrochemical reactions (1-5) show the existence of several oxidizing agents that may be responsible for corroding the iron contained in the steel that the columns are made of. These corroding agents are bicarbonate ions HCO_3^-, water, hydronium ion, and protonated amines [11].

Dissolution of iron (anodic reaction):

$$Fe \leftrightarrow Fe^{2+} + 2e^- \tag{1}$$

Reduction of the hydronium ion (cathodic reactions):

$$2H_3O^+ + 2e^- \leftrightarrow H_2O + H_2(g) \tag{2}$$

Reduction of bicarbonate ion:

$$2H_2CO_3^- + 2e^- \leftrightarrow 2CO_3^{2-} + H_2(g) \tag{3}$$

Reduction of undissociated water:

$$2H_2O + 2e^- \leftrightarrow 2OH^- + H_2(g) \tag{4}$$

Reduction of protonated amine:

$$2R_2NH_2^+ + 2e^- \leftrightarrow 2R_2NH + H_2(g) \tag{5}$$

The corrosive nature of CO_2-saturated amines which degrades the equipment, alongside the high cost of thermal regeneration and their volatility, are the main reasons for researching and developing other CO_2-absorbing solutions.

3.1. Corrosion test

Three solvents were studied in this paper: aqueous MEA 30%, ethaline ChCl:EG 1:2 molar ratio and ChCl:MEA 1:8. Tushar J. Trivedi and co-authors

[12] proposed a laboratory corrosion test of DES, which simulates one year of industrial use. Metal plates (stainless steel) were immersed in the selected solvents in a closed glass vessel. The vessels were then placed in an oven at 90°C, for 10 days. Six corrosion tests were done using this method: each solvent free of CO_2 (replicating the conditions inside an absorption column) and each solvent saturated in CO_2 (replicating the conditions inside the desorption column).

3.2. Degradation products and formation mechanisms

Oxidative degradation can be observed due to the appearance of compounds with different molecular weights than MEA. Table 1 lists some compounds that are formed [4], and the chemical mechanisms of formation will be explained in the following sections.

Table 1

Main degradation products of MEA [4]

Solvent	Degradation product	Molecular weight g/mol
MEA	Ammonia	17
	Formic acid	46
	Acetic acid	60
	2-oxazolidone	87
	N-(2-hydroxyethyl)formamide	89
	N-(2-hydroxyethyl)imidazole	112
	N-(2-hydroxyethyl)piperazin-3-one	144
	N-(2-hydroxyethyl)-2-(2-hydroxyethylamino)acetamide	162
	N,N’-Bis (2-hydroxyethyl)oxamide	176

Two main mechanisms are proposed for degradation: electron acceptance and hydrogen release. According to the literature, the mechanism of electron abstraction from nitrogen in the amino group by a reactive free radical (most likely a metal ion released by the corrosion phenomenon). The number of free radicals directly affects the rate of abstraction. The amine radical and the amine cation radical react forming an imine radical, after which it releases yet another a free radical to produce an imine.

The second mechanism is the extraction of hydrogen, from a cyclic amine structure, formed through hydrogen bonds. The resulting amine radical is transferred inside the structure and stabilizes by forming degradation products, mainly ammonia, aldehydes and aldehyde radicals. Hydrogen is most likely extracted from α-carbon. Bedell [13] proposed two pathways for hydrogen abstraction: the hydroxyl radical or the organo-peroxyl radical, in order to produce and form an organic radical (Fig. 1).

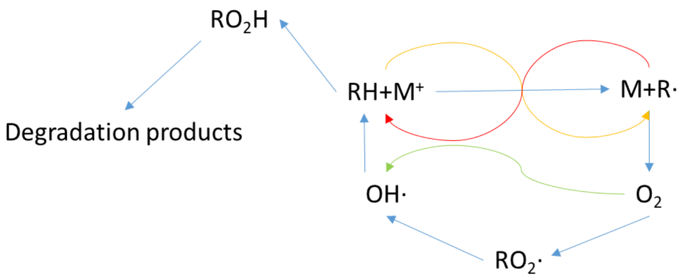

Fig. 1. Potential precursors for hydrogen extraction adapted from Bedell [13]

Carboxylic acids are the most common degradation compounds in amine-based solvents, as shown in Table 1. Aldehydes are self-oxidized in the presence of oxygen, forming peroxyacid, which then stabilizes in carboxylic acids. Carboxylic acids can be dissociated in basic solutions in order to obtain heat-stable amine salts.

3.3. Degradation analysis of DES using FTIR-ATR

Analysis of liquid monoethanolamine (MEA) samples was performed by Fourier transform infrared spectroscopy (FTIR), with total attenuated reflectance (ATR). The equipment used was a Shimadzu IRTracer-100 spectrometer and spectra processed using IRSolutions software from Shimadzu, as well as Origin Pro 2018 data processing software.

Each solvent spectra was compared to the spectra of the individual constituent of the DES. Each solvent was analyzed in all the usage stages: (a) fresh solvent, (b) solvent with captured CO_2, (c) fresh solvent after the corrosion test (abbreviated *solvent coroz)* and (d) solvent with captured CO_2, after the corrosion test (abbreviated *solvent+CO_2 coroz).*

3.3.1. MEA 30% solvent

Pure MEA has spectral bands characteristic of the amino $-NH_2$ group between the wavenumbers 3287-3293 cm^{-1}, as well as the hydrocarbon-ethyl, at 3355 cm^{-1} form the alcohol group, with its O-H bond. The C-H bond is present at 2957 and 2882 cm^{-1}. 1635-1638 cm^{-1} are vibration bands of O-H type bonds. Specific for amines is the signal at 1603 cm^{-1} $-H_2C-NH_2$ [14], in adition $-H_2C-OH$ with peaks at 1460 and 1358 cm^{-1}. C-N has a spectral band at a wavenumber of 1074 and C-O at 1026 cm^{-1}. **MEA 30%** (aqueous solution) shows weaker spectra due to the presence of water in the sample, while the spectral lines were similar to pure MEA having peaks at 3282, 1456, 1339, 1070, 1020 cm^{-1} (Fig. 2).

The reaction of MEA with CO_2 is known to form a carbamate compound. The next studied sample was **MEA 30% after CO_2 absorption**. In the 30% aqueous MEA solution CO_2 was bubbled to saturation. Two new signals specific to carbamate -HN-CO_2 appear at wavenumbers of 1337 and 1153 cm^{-1}. The imino-carbonate bond (-C-O) is present at 1496 cm^{-1} and at 1362-1361 cm^{-1}.

The **MEA 30% coroz** and **MEA 30% + CO_2 coroz** samples were subjected to the corrosion test. A decrease of the transmittance ca be observed in the area of 3200 cm^{-1} and at the same time the appearance of new signals around 2700 cm^{-1}. In the range of 1700-1200 cm^{-1}, new stronger signals appear and another clear, distinct peak is formed in the area of 1050 cm^{-1}. It is visible that some of the bands of the C-C, C-H and C-N from the 3200 cm^{-1} area have changed into various other type of bonds, having new peaks in the area 2884 cm^{-1} which are characteristic to aldehydes (-CHO); the 2350-2375 cm^{-1} area is more pronounced after the corrosion test, part of the "broadband ammonium" $C=N^+$-H, where carbon forms a double bond with nitrogen, becoming the ammonium ion. The formation of aromatic rings is not observed, the characteristic signals of the hydrocarbon cycles in the area 3010-3040 cm^{-1} are completely missing. On the other hand, alkenes were formed, the simple, saturated bond between C-C in ethanol, transforms into a double bond C=C, which is demonstrated the intense band of 1651-1650 cm^{-1}. The bands from 1400-1200 cm^{-1} suggest the appearance of nitro- and nitrosoderivatives, 1555 and 1370 cm^{-1} C-NO_2 and N-NO_2, 667 cm^{-1} associable with O-N=O and 1430-1458 cm^{-1} for N-N=O.

The wavenumber range 1700-1200 cm^{-1} can also be associated with $Fe(OH)_3$, which according to the IRSolutions software's database. This is plausible, due to the corrosion marks left on the stainless steel plate, used in the test. Metal ions act as a catalytist when degrading amines into carbamates.

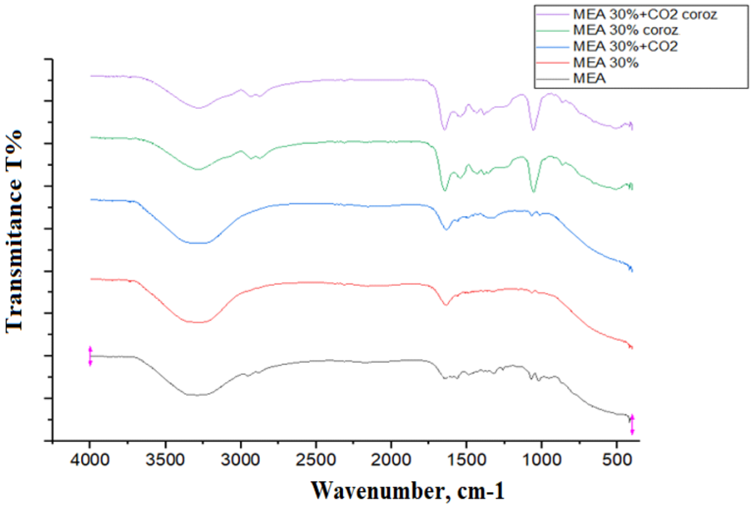

Fig. 2. FTIR spectra of MEA 30% degradation

The formation of ten primary degradation compounds (acids, ammonia and alkylamines) and seven secondary degradation compounds (HEGly, OZD, HEPO, HEF, HEA, HEI and BHEOX) was observed [15, 16]. FT-IR analyzes the types of chemical bonds in samples, which shouldn't be considered a complete identification of the compounds. This can be done by associating a mass spectrum, seen in the bibliographic studies mentioned in this paragraph.

3.3.2. Solvent DES ChCl:EG 1:2

Pure substances were initially studied: ethylene glycol and choline chloride. Ethylene glycol is an organic compound that contains two carbon atoms linked by a saturated chain, to which two hydroxyl groups are attached, resulting in a diol. The spectrum has a band at 3287 cm^{-1}, which is specific to the extension of the O-H bond [17]. As seen in Fig. 3, the O–H stretching vibration in the composition of the studied solvents changes to 3300 cm^{-1}, indicating that the O–H in the EG takes part in the formation of the hydrogen bond with the anion of choline chloride, specific to an eutectic solvent. The peaks at 2934 cm^{-1} and 2870 cm^{-1} belong to the asymmetric tensile vibrations of CH_2 and the symmetrical tensile vibration, respectively. The 1030 cm^{-1} and 1082 cm^{-1} bands are particular to the C–O and C–C skeletal vibration bands. The double bands 860 cm^{-1} and 881 cm^{-1} belong to the stretching vibrations -CH_2 group [18].

Choline chloride is a quaternary ammonium salt which contains a Cl^- anion that will act as a hydrogen bond acceptor. Choline chloride contains several functional groups, but few of them coexist after the formation of DES; the vibrational bands from 3217 cm^{-1} and the area 1200-880 cm^{-1} refer to a hydroxyl or amino group (first stretch of N-H, respectively, C-N^+ second symmetrical stretch), meanwhile vibrational bands at 3024-2845 cm^{-1} and 1481-1414 cm^{-1} refer to an alkyl group. This is the curvature of the CH_2 bond at 1481 cm^{-1}, which is a prominent group detected in all DES based on choline chloride [19-21].

The eutectic **ChCl:EG 1:2 (ethaline)** was studied in comparison with pure substances, choline chloride and ethylene glycol, respectively. Between 3600 and 3000 cm^{-1} the vibration of hydrogen bonds is observed, in various functional groups, such as O-H N-H, and / or O-H···O and/or N-H···O which denotes the existence of molecular aggregates specific to eutectic mixtures. Ethaline has a very wide and high intensity band, specific to intramolecular –OH bonds, and indicates the formation of new hydrogen bonds and / or molecular aggregates, with a maximum at 3300 cm^{-1}. It is also noticeable the formation of new H bonds by the formation of the absorption band from 3026 cm^{-1}, specific to the O-H···N-H and/or N-H···O type bonds. The appearance of the bands specific of the C-C-O-groups from 1083 cm^{-1} and from 1040 cm^{-1} and the HO···HN group from 953 cm^{-1} is also noteworthy.

Ethaline + CO_2. Carbamate has specific bands at 1319 and 1130 cm^{-1}, signals of -HN-CO_2. The imino-carbonate (-C-O) is present at 1479 cm^{-1}. Aldehyde bands (-CH=O) can be seen at 2872 and 2934 cm^{-1}, the ammonium cation bounded to -CH_2 can be detected at 1418 cm^{-1}, the symmetrical CO bond in the carboxyl group (-COO-) is present at 1269, 1238 and 1040 cm^{-1}.

Ethaline with and without CO_2 absorbed were tested for corrosion, similar to 30% MEA, using a stainless steel plate. **Ethaline coroz** and **ethaline+CO_2 coroz** do not show new bands when compared to the samples before the corrossion test. This highlights one of the biggest advantages of eutectic solvents, namely thermal stability over time. The solvents slightly changed color at the end of the test, probably because traces of $Fe(OH)_3$ have formed, supported by the signals that appeared in the range of 1200 cm^{-1}. Althought the signals are hardly visible probabliy due to the low concentration of Fe^{+3} ions and are also overlapped by other various organic compounds.

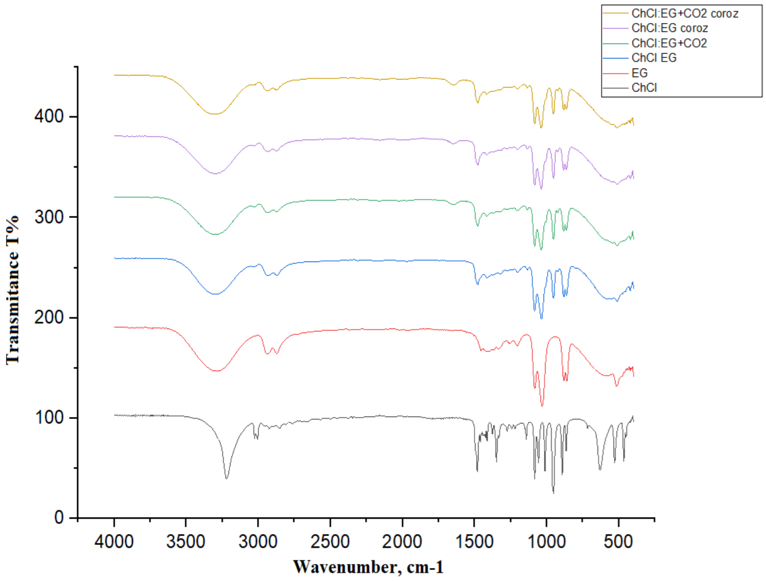

Fig. 3. FTIR spectra of ChCl:EG 1:2 degradation

3.3.3. *Solvent DES ChCl:MEA 1:8, CO_2*

The eutectic **ChCl:MEA 1:8** was compared with pure substances, choline chloride and monoethanolamine, respectively. The two constituents were discussed above, therefore the eutectic formed in a molar ratio of 1:8 - ChCl:MEA will be discussed. Both CO_2-free solvent and solvent having CO_2 absorbed were subjected to the corrosion test.

The vibrational bands of the hydroxyl groups are present in all 4 samples, the functional specific groups from ChCl, appearing at 3321-3337-3356 cm^{-1}. Regarding the samples with captured CO_2, they have the same stable band at 3337 cm^{-1}, while ChCl:MEA before and after the corrosion test show slight variations of the OH groups. The bond of the ammonium ion is highlighted at the wavenumbers 864 and 866 cm^{-1}, respectively. In the case of DES with and without absorbed CO_2, both peaks were equally stable even after the corrosion test. The alkyl group shows an IR band at 2951 cm^{-1} for unused ChCl:MEA and **ChCl:MEA coroz**, as well as at 2934 cm^{-1} and 1475-1477 cm^{-1}. Such a band is additionally found at 1419-1420 cm^{-1} for CO_2-free ChCl:MEA and ChCl:MEA coroz. Both bands are specific to eutectic solvents based on choline chloride.

Amino $-NH_2$ spectral band is more evident in the case of **ChCl:MEA+CO_2** and **ChCl:MEA+CO_2 coroz** at the wavenumber 3282 cm^{-1}, due to the formation of carbamate. 2876 cm^{-1} and 2882 cm^{-1} are signals of the C-H bond from DES with CO_2, but absent in the case of ChCl:MEA coroz, most likely because the bond changed to another type. It should be noted that the captured CO_2 samples have been stable for a long period of time, surprisingly, surpassing their CO_2-free counterparts. 1645-1653 cm^{-1} are vibration bands of the O-H type bonds, with slightly lower values for the samples containing carbamate. The spectral band for the C-N bond is found in the range 1063-1072 cm^{-1}, and the C-O bond at 1009-1024 cm^{-1}. The tensile vibrations of C-(O)-O are noticed at 1312-1319 cm^{-1}, and in the case of the samples with captured CO_2, the tension vibrations of C-O shifted to 1155 cm^{-1}.

No new chemical bonds were observed based on Fig. 4. Comparing the samples from the corrosion test with the unused solvents, no significant differences were noticed. This fact enforces one of the main advantages of the eutectic solvents, namely, their high thermal stability.

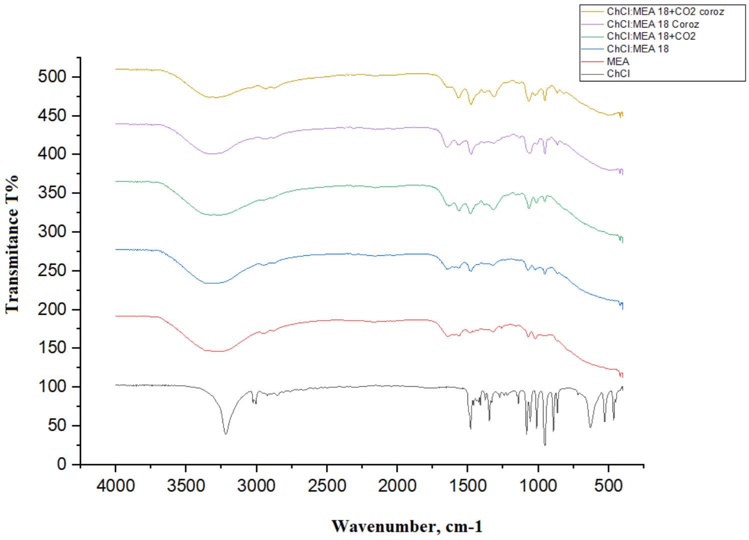

Fig. 4. FTIR spectra of ChCl:MEA 1:8 degradation

3.4. Study of the corrosion of metal plates using XRFS

X-ray fluorescence spectroscopy (XRFS) is a method that qualitatively and quantitatively determines the composition of a material, as well as for measuring the thickness of the layers that cover the samples. XRFS is based on the emission of X-rays specific to a material, after it was irradiated with X-rays that induced fluorescence. Stainless steel plates were immersed into the six distinct samples, heated in the laboratory oven and analyzed (triplicate testing). Using Vanta's portable XRF Olympus analyzer, the main components on each metal plate were determined and compared to an untreated plate. Both variants were studied, with and without absorbed CO_2, replicating the conditions inside of the absorption and desorption reactors and the effect they have on the metal they come into contact with.

In **MEA 30%** case (Fig. 5), after the 10 days of testing (equivalent to 1 year of use), there was a 0,09% increase in the percentage of Fe deposited on the surface of the plates and a 0,058% increase in Cr, but also a 0,02% decrease in Mn and 0,028% decrease in Ni. For MEA 30% with absorbed CO_2, there are 3 detectable increase of deposits on the surface: Cr by 0,037%, Mn by 0,02% and Fe by 0,103%, while the Ni decreased by 0,03%.

The next solvent studied was **ChCl:EG 1:2** (Fig. 5), also with and without absorbed CO_2. Within the deposits there were 3 metal components that increased their presence on the surface: Cr (0,1%), Fe (0,13%) and Mn (0,01%). Ni had a slight decrease of 0,057%. The same trend was maintained after CO_2 absorption, namely increased values for Cr (0,097%), Mn (0,043%) and Fe (0,1%).

The third type of solvent chosen was **ChCl:MEA 1:8** (Fig. 5). A higher percentage of Fe compared to the previous solvent is observed, by 0.193% compared to the untreated plate. Increases are also recorded for Cr (0,07%) and Mn (0,01%), and as before, Ni decreased (0,073%). After the absorption of CO_2, the solvent becomes less corrosive, demonstrated by the percentages of Fe on the surface: +0,11% compared to 0,19% (a reduction of about 58% between the two solvents). This phenomena can be explained by the formation of carbamate, that reduced the amount of MEA, implicitly the corrosion effect on the metal plate.

The main component in stainless-steel is iron, which makes up aproximately 70%. For the 3 eutectic solvents, the amount of Fe deposited on the surface of the plate as a result of corrosion is lower after CO_2 is absorbed, and slightly higher in the reference case – MEA 30% aq. Another noticeble deposit is Cr. Mn is at the limit of detection for XRF. In all cases, the Ni content seems to decrease, although this might not be the case because the nickel cointaining areas are coated by Cr and Fe deposits.

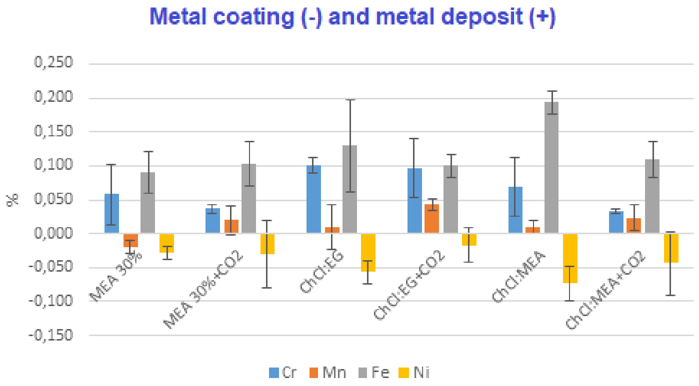

Fig. 5. Summary of metal coating or deposit from the plates

4. Conclusions

Deep eutectic solvents can be a viable solution in capturing CO_2 from post-combustion gases, due to their high chemical and thermal stability and also, their reduced corrosive effect on the equipment.

The present work simulated one year of use of the reference solvent, MEA 30% aq. and two DES, ethaline ChCl:EG 1:2 and ChCl:MEA 1:8. MEA 30% suffered transformations, which can be seen in the bands specific to "broadband ammonium" C=N, where the carbon and the nitrogen form a double bond, creating the ammoniu cation. From the corroded stainless steel plate, traces of Fe^{3+} could be detected, adding a catalyst to the system that initiates oxidation reactions.

ChCl:EG 1:2 and ChCl:MEA 1:8 did not exhibit new bands after the corrosion test in comparison to the unused samples. The samples with absorbed CO_2, showed no signs of degradation, just traces of carbamate, the main product of the absorption of CO_2 in amines. MEA which is inherently corrosive, has a mitigated effect in the eutectic mixture. This highlights the main advantage of the deep eutectic solvents for CO_2 capture from post-combustion gases, their thermal and chemical stability over time.

The conclusion of this corrosion test is that the selected eutectic solvents have a minimal corrosion effect on the equipment, with material losses being around 0.1-0.2% per year. Another important aspect is the metal alloy used for the equipment, which has to be resistant to the inherent corrosion effect of solvents such as MEA 30%.

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