

STABILIZATION OF LEACHATE IN AGGREGATES MADE WITH PLASTIC FROM WEEE

Estabilización de lixiviados en agregados elaborados con plásticos de RAEE

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ABSTRACT

A recycled plastic aggregate (RPA) was developed using the core-shell strategy, where the core is the plastic fraction of Waste of Electrical and Electronic Equipment (WEEE) and a cement matrix with stabilizing additives acts as the shell. The amount of brominated flame retardant (mainly tetrabromobisphenol-A) leached in curing water of RPAs was quantified using extraction with an organic solvent and gas chromatography methods (CG-FID). A clear relationship can be established between the characteristics of the stabilizing additive used and the amount of tetrabromobisphenol-A and bisphenol-A leached. The additive used was activated carbon, which in a manufacture scale may be provided by different suppliers with different mesoporous characteristics, which can be easily determined by the iodine number. The analysis proposed can be an effective way to determine if a particular activated carbon can be used as stabilizing additive in the production of RPAs with the developed technology.

Palabras clave: gestión de residuos, agregado de plástico reciclado, retardantes de llama bromados, plásticos de RAEE

RESUMEN

Se desarrolló un agregado de plástico reciclado (APR) usando la estrategia de núcleo-coraza en que la fracción plástica de residuos de aparatos eléctricos y electrónicos (RAEE) es el núcleo o corazón y una mezcla de cemento y aditivos estabilizantes actúa como coraza. La cantidad de retardantes de fuego bromados (principalmente tetrabromobisfenol-A) lixiviados en agua para el curado de APR se mediante extracción con solventes orgánicos y cromatografía gaseosa (CG-FID). Se logró establecer una relación clara entre las características del aditivo estabilizante y la cantidad de tetrabromobisfenol-A y bisfenol-A lixiviados. Se utilizó como aditivo carbón activado, que en producción de gran escala puede ser provisto por diferentes proveedores

comerciales con diferentes características de mesoporo. Esta característica puede evaluarse fácilmente mediante el número de yodo y es determinante en la capacidad de estabilización del carbón activado. El análisis propuesto puede utilizarse de manera efectiva con la tecnología desarrollada para determinar la capacidad estabilizante del carbón empleado como materia prima en la producción de RPA.

INTRODUCTION

Waste from electrical and electronic equipment (WEEE) grew significantly over the last 20 years (Tan et al. 2017). The plastic fraction of WEEE (WEEEP) represents about 20 % of the total mass (Wäger et al. 2012). A real sample of WEEEP is a complex mixture of different types of polymers; yet, three types are predominant, acrylonitrile butadiene styrene (ABS, 30-35 %), high impact polystyrene (HIPS, 20-25 %) and polypropylene (20-30 %). Also, polycarbonate (PC) is present (Schlummer et al. 2007, Dimitrakakis et al. 2009a, Wäger et al. 2012, Peeters et al. 2014, Maris et al. 2015). It is widely known that this plastic contains heavy metals (Tamaddon and Hogland 1993, Dimitrakakis et al. 2009b, Stenvall et al. 2013), brominated flame retardants (BFRs) (Schlummer et al. 2005), polymer decomposition substances, and other hazardous compounds.

BFRs are persistent organic pollutants that can be accumulated and detected in humans and the environment (Meironyte et al. 1999, Covaci et al. 2006, Law et al. 2006). Over the last decades, concerns have been raised over the toxicity of some BFR compounds and their risk to human health (Darnerud 2003, Dunnick et al. 2017). Currently, it is difficult to recycle WEEEP and it is treated by incineration or landfilling (Ilankoon et al. 2018).

However, WEEEP could be used to develop novel recycled materials (Schlummer et al. 2006, Buekens and Yang 2014, Luhar and Luhar 2019), in particular novel building materials (Siddique et al. 2008, Saikia and de Brito 2012, Gu and Ozbakkaloglu 2016, Senthil-Kumar and Baskar 2018). An example of these are the recycled plastic aggregates (RPA) (Hannawi et al. 2010, Lakshmi and Nagan 2010, 2011, Wang and Meyer 2012, Alqahtani et al. 2014, 2017a, b, Nowek 2016), and the use of WEEEP as coarse aggregate for concrete (Senthil-Kumar and Baskar 2015a, b, c) and fiber for concrete mixture (Gu and Ozbakkaloglu 2016).

It is particularly important to analyze the impact of both contaminants (heavy metals and polybrominated organic compounds) in the final recycled material. In this way, Senthil-Kumar et al. (2016) and our

research group considered the presence of metals in the leaching of Portland cement base materials made with WEEEP (Peisino et al. 2019). Also, we recently published a study about the behavior of the BFRs present in WEEEP (specifically tetrabromobisphenol-A [TBBPA]), in a Portland cement system (Gómez et al. 2020). We have determined that the hazardous organic contaminant (TBBPA) leached in a higher proportion when WEEEP was in contact with the basic medium of the Portland cement. However, this effect was solved with the use of activated charcoal (AC) as a stabilizing additive.

AC are composed of carbonaceous matter with a porous structure. They are generally manufactured in two stages: firstly by carbonization (pyrolysis) of the organic matter (essentially wood of different types), and then by an activation process (Yang 2003). The pyrolysis stage removes elements other than carbon, producing a porous material. The activation stage is necessary in order to increase the adsorption capacity of the material. There are many kinds of activation procedures that can be performed: physical activation (in presence of water vapor and air under pressure) or chemical activation (usually by treatment with phosphoric acid) (Yin et al. 2007). AC are particularly attractive for the capture of inorganic and organic contaminants due to their porosity (pore size ranging from 4.5 to 60 Å), good adsorption capabilities, relatively low production cost, and large specific surface area (300-4000 m²/g) (Yang 2003). In addition, it is possible to modify the adsorption characteristics of activated carbons changing the raw material to be carbonized and by optimization of the carbonization process (Laine and Yunes 1992) and activation steps (Yin et al. 2007).

In this work we present the results on the deepening of the analysis of activated charcoal types and dosage. A core-shell approach was applied, where the particles of plastic wastes were covered with a mixture of masonry cement and activated charcoal. We evaluated the leaching of TBBPA and bisphenol-A (BPA) in the curing water of the different formulations of RPAs. To achieve this, the extracts were analyzed by gas chromatography (GC) to determine the concentration of organic compounds leached and

so evaluate the capability of the different activated charcoals to stabilize pollutants.

MATERIALS AND METHODS

Reagents and materials

WEEEP (WP) was supplied in crushed form by a local recycling company which collects and processes all types of waste of electric and electronic equipment. The apparent specific gravity of the used WEEEP was 510 kg kg/m³. Masonry cement (MC) from Holcim was employed. Powder activate charcoals were obtained from different suppliers and were used as received. CAE 061, MMF, CAE Ultra and Clarisorb B were obtained from Clarimex; Merck was obtained from Merck. Powder vegetal charcoal ("Vegetal") was obtained from a local supplier. In order to obtain a homogeneous sample, the carbon was screened with a 75- μ m sieve (200 mesh).

Milli-Q water was employed for the preparation of RPAs and to carry out the curing of these materials. Analytical grade *n*-hexane, ethyl acetate, and hydrochloric acid (HCl, 37 %) were used as they were received from the supplier. For iodine number determination the following reactants were used: potassium iodate (KIO₃, GR for analysis), potassium iodide (KI, GR for analysis), iodine (I₂, GR for analysis) sodium thiosulphate (Na₂S₂O₃, GR for analysis), and starch.

TBBPA was obtained by column chromatography isolation in our laboratory with 99 % purity determined by GC-MS. For quantification, two standards were employed: docosane \geq 99 % (C₂₂H₂₄), and 9-bromoanthracene \geq 99 % (C₁₀H₁₅Br), both from Fluka.

RPA manufacture

The following procedure was used to manufacture the core-shell RPA, as described in a previous work (Gómez et al. 2020):

- First, the WP (core) was placed in the mixer and water was added to wet the plastic particles.
- Next, the cementitious mixture for the shell was added slowly and mixed by hand with enveloping movements.
- The shell was built in three layers, with a waiting time of 24 h between layers. In addition, particles were screened with a 4.8 mm sieve (4 mesh) between each layer.
- Finally, RPAs were cured by water immersion for 14 days.

Twenty-seven different RPAs were prepared. All samples were made using 250 g of WP covered with a mixture prepared with 300 g of MC, different amounts and types of AC and 100 mL of water (**Table I**).

TABLE I. RECYCLED PLASTIC AGGREGATES (RPAs) FORMULATIONS AND COMPOSITIONS¹

Entry	Sample	Carbon type	Amount of carbon ² (g, %)
1	WP@MC	—	—
2	WP@MC:CAE-1	CAE 061	(6.25, 1.1)
3	WP@MC:CAE-2	CAE 061	(12.50, 2.2)
4	WP@MC:CAE-3	CAE 061	(18.75, 3.3)
5	WP@MC:CAE-4	CAE 061	(25.00, 4.3)
6	WP@MC:MMF-1	MMF	(6.25, 1.1)
7	WP@MC:MMF-2	MMF	(12.50, 2.2)
8	WP@MC:MMF-3	MMF	(18.75, 3.3)
9	WP@MC:MMF-4	MMF	(25.00, 4.3)
10	WP@MC:CAEU-1	CAE Ultra	(6.25, 1.1)
11	WP@MC:CAEU-2	CAE Ultra	(12.50, 2.2)
12	WP@MC:CAEU-3	CAE Ultra	(18.75, 3.3)
13	WP@MC:CAEU-4	CAE Ultra	(25.00, 4.3)
14	WP@MC:CLARI-1	Clarisorb B	(6.25, 1.1)
15	WP@MC:CLARI-2	Clarisorb B	(12.50, 2.2)
16	WP@MC:CLARI-3	Clarisorb B	(18.75, 3.3)
17	WP@MC:CLARI-4	Clarisorb B	(25.00, 4.3)
18	WP@MC:MERCCK-1	Merck	(6.25, 1.1)
19	WP@MC:MERCCK-2	Merck	(12.50, 2.2)
20	WP@MC:MERCCK-3	Merck	(18.75, 3.3)
21	WP@MC:MERCCK-4	Merck	(25.00, 4.3)
22	WP@MC:VEG-1	Vegetal	(6.25, 1.1)
23	WP@MC:VEG-2	Vegetal	(12.50, 2.2)
24	WP@MC:VEG-3	Vegetal	(18.75, 3.3)
25	WP@MC:VEG-4	Vegetal	(25.00, 4.3)
26	WP@MC:VEG-19	Vegetal	(125.00, 18.5)
27	WP@MC:VEG-31	Vegetal	(250.00, 31.3)

¹All the RPAs were prepared with 250 g of WEEEP and 300 g of masonry cement. The shell was prepared in three layers. Between layers the waiting time for cement setting was of 24 h. In addition, between each layer, particles were screened with a 4.8 mm sieve (4 mesh).

²The percentage is relative to the amount of WEEEP.

Chemical analyses

Quantitative analyses of organic compounds were performed by gas chromatography using a Shimadzu GC-2010 gas chromatograph with flame ionization detector, equipped with a VF-5ms column (30 m \times 0.25 mm \times 0.25 μ m) using N₂ as gas carrier. Quantification of TBBPA was made using the internal standard calibration method ($r^2 > 0.99$)

with authentic samples. Limit of detection (LOD = 84 mg/L) was calculated using the standard deviation of the residuals from the calibration curve (three times this standard deviation). Two internal standards were used, thus the LOD informed was a mean of these two calibrations. Limit of quantification (LOQ = 279 mg/L) was calculated using the standard deviation of the residuals from the calibration curve (10 times this standard deviation).

Pore water from the cured process of the RPAs was obtained with the following procedure: immersion of RPA containing 250 g of WP in 500 mL of milli-Q water for 14 days at room temperature (25 °C) without stirring. Then the RPA (solid) was filtered and the solution was acidified with concentrate hydrochloric acid at pH = 2. The aqueous phase was extracted with *n*-hexane (4 × 125 mL), the organic phase was dried with sodium sulphate, and the solvent was evaporated in vacuum. Standards were added to the organic phase and it was dissolved in ethyl acetate (10 mL); the organic compounds (TBBPA and BPA) were quantified by GC-FID using an internal standard method.

Iodine number determination was carried out for all carbon samples by a titration method described in the ASTM D 4607 standard (ASTM 2006). The values are shown in **table II**.

TABLE II. IODINE NUMBER AND SPECIFIC SURFACE AREA OF THE ACTIVATED CARBON SAMPLES.

Entry	Sample	Manufacturer	Iodine number ¹	S _{BET} ² (m ² /g)
1	CAE 061	Clarimex	552	544
2	MMF	Clarimex	764	755
3	CAE Ultra	Clarimex	792	783
4	CLARISORB B	Clarimex	794	785
5	Merck	Merck	777	768
6	Vegetal	Local supplier	124	112

¹Determined according to the ASTM D 4607 standard (ASTM 2006).

²Specific surface area (S_{BET}) estimated from the iodine number (IN): S_{BET} = (0.9946 × IN) – 4.91 (Mianowski et al. 2007).

RESULTS AND DISCUSSION

RPAs manufacturing

The main polymers in WEEEP samples were HIPS (34.5 %), ABS (62.0 %) and ABS/PC (2.2 %), which were determined by FT-IR in a previous report (Peisino et al. 2019). Better results in term of shapes

and size homogeneity were achieved when three layers of cementitious covering were applied (Gómez et al. 2020). Then, RPAs were prepared according to Gómez et al. (2020). The identity of the activated charcoal used, and the dosage were modified to obtain samples that allowed establishing the most effective stabilization dosage for each activated carbon assayed.

Iodine number

The iodine number (IN) is the mass of iodine (in milligrams) consumed by 1 g of a chemical substance, commonly used to estimate the insaturation grade of oils and fatty acids, but it is also related to the porous characteristics of sorbents materials such as activated carbon, and fly ash, among others (Mianowski et al. 2007). Although the sorbent capability of a substance depends on variables such as porosity, pH, zeta potential, and temperature, for the proposed use conditions of the activated carbon, it is more related to porosity, since pH would be determined by the cement matrix and temperature is difficult to control and monitor in the production of construction materials (Mianowski et al. 2007).

In addition, a specific surface area can be estimated from the IN values, according to Mianowski et al. (2007).

As can be observed on **table II**, it is possible to divide the activated carbons employed in three categories: low IN (Vegetal), medium IN (CAE 061) and high IN (the rest of the carbons tested). This means that Vegetal charcoal is the less porous, while Merck, MMF, CAE Ultra and Clarisorb B had the highest porous character.

Chemical analysis

TBBPA has two ionizable aromatic hydroxyl groups (pK_{a1} = 7.5 and pK_{a2} = 8.5) (Bergman et al. 2012). When TBBPA is in contact with cement shell, which is a well-known strong base (Birchall et al. 1978), it reacts immediately to form the anionic species of TBBPA (**Fig. 1**), thus increasing leaching to the aqueous phase. The same behavior applies to BPA, which is also a phenol.

Table III shows the concentrations of organic compounds for each leachate measured by CG-FID analysis. **Figure 2** shows data from **table III** represented in a graphic bar. From the analysis of **table III** and **figure 2** it appears that all activated carbons tested, in the proposed dosages (1.1 to 4.3 %), produce leachates below the LOD for TBBPA. This is consistent with the high value and specific surface area estimated by IN for these carbons. Activated carbon with a specific surface area of 896 m²/g determined

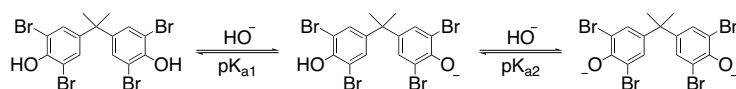


Fig. 1. Acid-base equilibrium of tetrabromobisphenol-A (TBBPA).

TABLE III. ORGANIC COMPOUND EXTRACTION OF DIFFERENT RE-CYCLED PLASTIC AGGREGATES (RPAs)¹.

Entry	Sample	[TBBPA] ²	TBBPA ³	BPA ⁴
1	WP@MC	(105±2)	(41.8±0.7)	1.00
2	WP@MC:CAE-1	< LOD ⁵	< LOD	0.48
3	WP@MC:CAE-2	< LOD	< LOD	0.63
4	WP@MC:CAE-3	< LOD	< LOD	0.90
5	WP@MC:CAE-4	< LOD	< LOD	0.48
6	WP@MC:MMF-1	< LOD	< LOD	0.60
7	WP@MC:MMF-2	< LOD	< LOD	0.45
8	WP@MC:MMF-3	< LOD	< LOD	0.33
9	WP@MC:MMF-4	< LOD	< LOD	0.33
10	WP@MC:CAEU-1	< LOD	< LOD	0.48
11	WP@MC:CAEU-2	< LOD	< LOD	0.29
12	WP@MC:CAEU-3	< LOD	< LOD	0.27
13	WP@MC:CAEU-4	< LOD	< LOD	nd ⁶
14	WP@MC:CLARI-1	< LOD	< LOD	0.46
15	WP@MC:CLARI-2	< LOD	< LOD	0.37
16	WP@MC:CLARI-3	< LOD	< LOD	0.22
17	WP@MC:CLARI-4	< LOD	< LOD	0.37
18	WP@MC:MERCK-1	< LOD	< LOD	0.41
19	WP@MC:MERCK-2	< LOD	< LOD	0.32
20	WP@MC:MERCK-3	< LOD	< LOD	0.04
21	WP@MC:MERCK-4	< LOD	< LOD	0.03
22	WP@MC:VEG-1	(100.0±0.3)	(40.0±0.1)	1.06
23	WP@MC:VEG-2	(85.0±0.5)	(34.0±0.2)	0.88
24	WP@MC:VEG-3	(63.5±0.5)	(25.4±0.2)	0.85
25	WP@MC:VEG-4	(58.3±0.5)	(23.3±0.2)	0.86
26	WP@MC:VEG-19	< LOQ ^e	< LOQ	0.70
27	WP@MC:VEG-31	< LOD	< LOD	0.15

¹Water for curing the RPA. Immersion of RPA containing 250 g of WEEEP in 500 mL of milli-Q water for 14 days at 25 °C without stirring was performed.

²Concentration of TBBPA (x 10¹ mg/L) in the concentrated solution (total volume of 10 mL).

³TBBPA leachate from samples in mg_{TBBPA}/kg_{WEEEP} (mg/kg).

⁴BPA leachate from samples relative to WP@MC.

⁵LOD = 84 mg/L, LOQ = 279 mg/L.

⁶Not determined.

by BET was one of the most effective sorbents used for the removal of BPA from aqueous solutions in a study carried out by Zhou et al. (2012). This is in a good agreement with the efficiency that we obtained for activated carbons with specific surface area values between 500 to 800 m²/g for the leaching of TBBPA and BPA in pore waters. Taking into account these results, we propose that a dosage of 1.1 % for all the activated carbons, which has an IN greater than

500, is effective for the stabilization of TBBPA and to prevent its leaching to cure waters, and eventually to the environment.

On the other hand, it can be observed that the non-activated vegetal charcoal does not perform as well as other carbons, which is totally in agreement with the low IN and specific surface area shown in **table II**, and with the fact that this charcoal lacks an activation process.

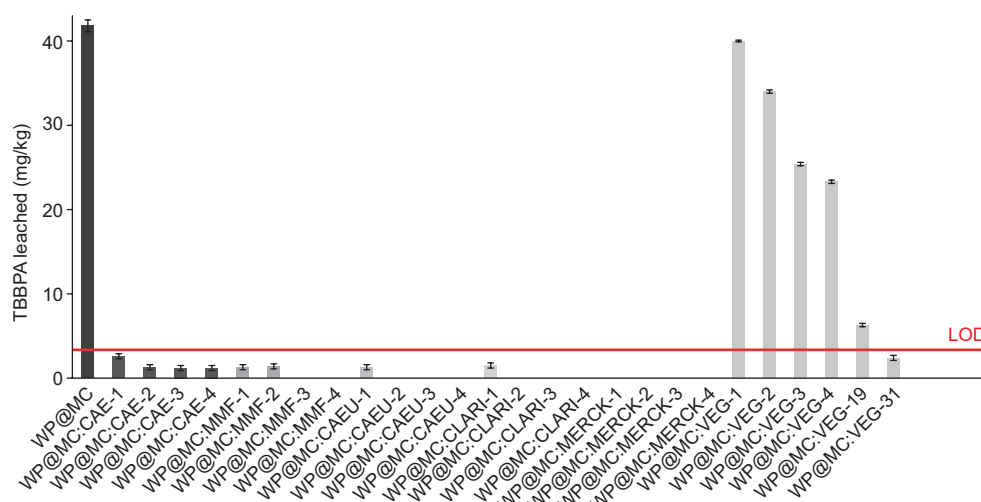


Fig. 2. Tetrabromobisphenol-A (TBBPA) leached in curing water of the different RPA.

Considering these findings, it can be said that for charcoals with low IN and specific surface area (IN lower than 150), an effective dosage in the production of RPAs must be of at least 31.3 %, which can be seen in more detail in **figure 3**.

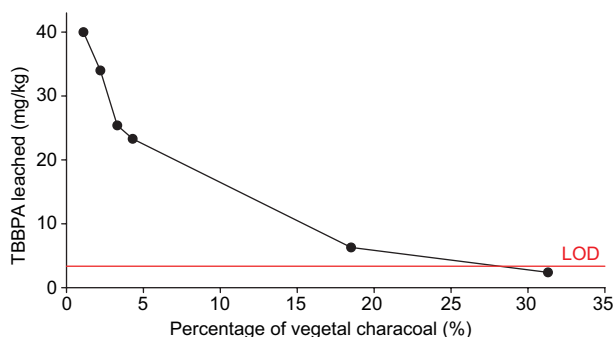


Fig. 3. Tetrabromobisphenol-A (TBBPA) leached in curing water of the different RPA covered with increasing percentages of “Vegetal” charcoal.

Figure 4 shows a relative quantification of BPA leached from the curing waters. The general tendency for the stabilization of BPA, the amount of activated carbon used in the production of RPAs is similar to that observed in TBBPA. The greater the dosage, the lower the BPA leached. However, it is important to notice that while all activated carbons

show similar behaviors, the Merck carbon proved to be more effective for the stabilization of BPA, though its IN or specific surface area is not the largest of all tested carbons. This could be evidence that, to some degree, other parameters such as the zeta potential or temperature can affect the efficiency of coal as a stabilizing agent. Still, it has been proven that IN is a robust parameter to anticipate the performance of a given carbon for stabilizing BFRs in the production of RPAs.

For the use of non-activated vegetal charcoal, the analysis of BPA leaching in pore waters was similar to the one observed for TBBPA. The amount of BPA leached in dosages of 1.1 to 18.5 % is considerable, but it can be said that in the dosage of 31.3 % stabilization of BPA is efficient.

CONCLUSIONS

In this work we used several activated carbons to produce RPAs with the plastic fraction of WEEE. Through chemical analysis and GC-FID we were able to measure the amount of TBBPA and BPA leached.

The IN was determined for each type of activated carbon used. Based on our findings, we can state that all carbons assayed with an IN higher than 500 can be used in a proportion of 1.1 % in the dosage for the production of RPAs with an effective stabilization of the organic contaminant present in the polymer matrix (BFR). Even though the IN is not related directly to the sorbent capability of the

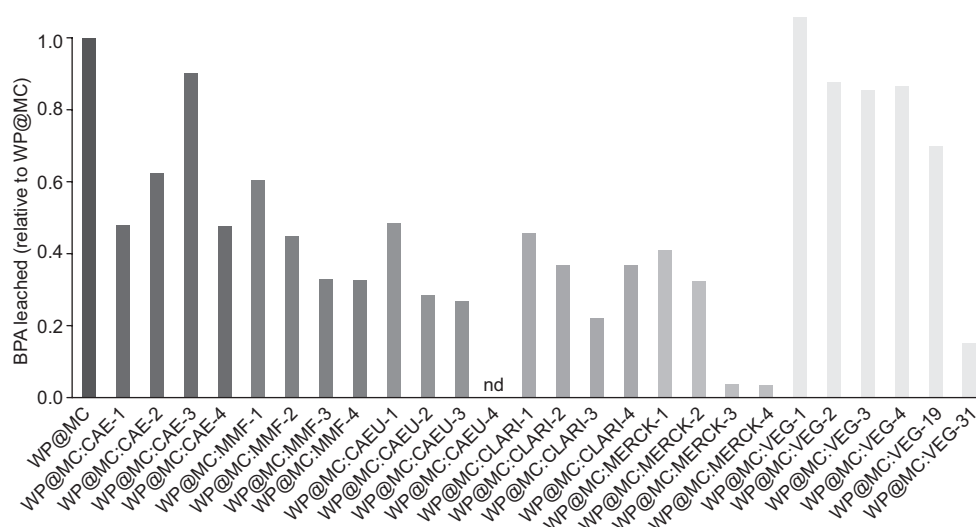


Fig. 4. Relative amount of bisphenol-A (BPA) leached in curing water of the different RPAs.

substance, but rather to its porosity, this index can be useful to predict the dosage needed for using a carbon not previously assayed, only by its simple and rapid determination.

Although vegetal non-activated charcoal needs to be added in a higher percentage (31.3 %) due to its low porosity (determined by the IN), this material can be easily obtained from a traditional charcoal factory. It is important to notice that charcoal was not subjected to an activation process, so it was unnecessary to use a power demanding processes like vapor treatments or acids. For this reason, the use of charcoal represents a more environmentally friendly, robust, and less expensive option for local ventures or cooperatives that want to implement the new proposed technology.

In further experiments we will study the effect of CA in the setting cement reaction. This will be crucial to determine the feasibility of using vegetable carbon in such high dosages.

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