Abstract

A practical and efficient treatment of PCBs (polychlorinated biphenyls) in transformer oil by a chemical dechlorination process has been reported. The transformer oil containing commercial PCB mixtures Sovtol-10 was treated by the required amounts of PEG 400 (polyethylene glycol 400) and powdered potassium hydroxide (KOH), along with different reaction times. The reaction of PEG with PCBs under basic condition produces arylpolyglycols and potassium chlorine. The relative efficiencies of PCB treatment process were assessed in terms of destruction and removal efficiency (DRE, %). Under the experimental conditions of KOH/PEG molar ratio 1.2:1, KPEG/oil 22:1 and three hours of reaction at 90 °C, average DRE of PCBs was approximately 99 %, showing completely removal of PCBs containing 5-7 chlorines. In the sample remain the congeners 52 and 44 that are not reported as toxic by the WHO (World Health Organization). With the previously reported conditions the scale up of the process was realized.

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Keywords: PCBs; dechlorination; KPEG process; transformers oils; design
1. Introduction

PCBs are chemical substances which are persistent, bioaccumulative, and pose a risk of causing adverse effects to human health and the environment. They can be transported over long distances, and have been detected in the furthest corners of the globe, including places far from where they were manufactured or used. While manufacture of PCBs has reportedly ceased, the potential or actual release of PCBs into the environment has not, since significant quantities of existing PCBs continue in use or in storage. [1]

Until now, no technology for the treatment of electrical equipment containing PCBs is available in Cuba. As a result of the ever increasing stocks of these equipments being taken out of service, the national strategy has been the temporary confinement under specific criteria that ensures the lowest risks possible to health and the environment. [2] The evaluation of source materials indicates that the priority in waste to be treated in Cuba is likely to be PCBs liquid. This is the reason why we will focus on evaluating the chemical deshalogenation technology.

According to an inventory carried out for United Nations Environment Programme (UNEP) about of worldwide PCB destruction capacity, there are 42 facilities from Africa, Asia, Australia, Europe, Latin America and the Caribbean, and North America that dispose PCBs and/or offer PCB destruction technologies and that facilities for PCBs destruction are mainly located in developed countries. [3]

High temperature incineration is the most often used proven process for the destruction of PCBs. Incineration, however, often emits more toxic compounds if is not carefully controlled. Polychlorinated dibenzodioxins and dibenzofurans have both been observed in the combustion of PCBs. [4] For that reason different methods for the destruction of PCBs have been proposed and include wet-air oxidation [5], super or supra-critical oxidation [6, 7], photolysis in the presence of hydrogen donors and oxidants [8], electrolytic reduction [9], biologic treatment [10, 11] and a number of other chemical methods, based on laboratory experiments only. Moreover, these methods typically involve one or more drawbacks, such as the use of expensive reagent, inert atmospheres, extensive temperature control, complex apparatus, substantial energy consumption and the like.

Among non-destructive decontamination processes (selective PCB removal), those based on the reaction between PCBs and PEG alkoxide appear as most promising from an industrial point of view. A group of reagents generically referred to as “APEG” (alkali metal polyethylene glycolate) has been developed.

Brunelle and Singleton [12] carried out an extensive study of Arochlor 1260 dechlorination in such non-polar media as heptane and toluene. They found that PCBs react with polyethylene glycols and potassium hydroxide under mild conditions, following a simple nucleophilic substitution by a polyethylene glycol alkoxide, probably occurring in the glycol phase. De Filippis [13, 14] applied the same chemical process to remove PCBs from contaminated dielectric and lube oils, concluding that the former are easier to dehalogenate. A first-order kinetic was observed for each PCB as well as with respect to the PEG concentration. The reaction was also affected by the KOH/PEG ratio. More recently, the influence of temperature, ultrasounds, polyethylene glycol base (PEG) and base type were studied for Cafissi [15]. They found that dechlorination of PCBs by chemical treatment is effective at moderate temperature (90-100 °C) and is strongly dependent on the nature of matrix oil. In addition, the ultrasound can improve the process efficiency by making operation condition less severe.

In previous investigations, the KPEG process using pellet KOH was studied. Average DRE of 93.7 % was obtained under the following conditions: PEG 400, of 30 and KOH/PEG mol ratio 2 for 2 hours of...
reaction. The ultrasound initially increases DRE of PCBs by 8% after 10 min of reaction time. After a reaction time of 120 min no difference in DRE was measured when using ultrasound.

The aim of the present work was to study the influence of KOH/PEG mol ratio, KOH/oil weigh ratio and ultrasound in the DRE (destruction removal efficiency) of Sovtol-10 using powdered KOH. The levels for these variables were changed. A preliminary kinetic study of the removal of PCBs and TCB from Sovtol-10 at 90°C was realized. A technology to pilot-plant scale starting from laboratory experimental results was designed. This proposal could be useful for developing countries.

2. Experimental

2.1. Materials

Experiments were carried out with transformer oil known as Sovtol-10 (it is a mixture of congeners with mainly four and five atoms of chlorine and trichlorobencenes) recovered from industrial transformers.

2.2. Chemical used

All chemicals (polyethylene glycol (PEG 400); powdered potassium hydroxide (KOH) and n-hexane) were analytical grade and provided by Fluka. Standard Aroclor 1254; 1, 2, 4-TCBZ; 1, 2, 3-TCBZ and 1, 3, 5-TCBZ and the CEN PCB Congener Mix-1 were purchased from Supelco. To calculate relative retention times, the congener 52 (2, 2, 5, 5'-tetrachlorobiphenyl) was used as “internal standard”.

2.3. Analytical methods

The PCBs determination was carried out using a Chrompack CP 9002 gas chromatograph (GC) with flame ionization detector (FID). The separation of congeners was done in 30 m x 320 μm x 0.25 μm 5% phenyl 95 % dimethylpolixilosane capillary column (HP-5). The GC experimental conditions were as follows: helium as carrier gas; injection temperature, 250 °C; detector temperature, 300 °C. The GC oven program was the following: initial temperature 110 °C, retained for 2 min, then increased at a rate of 5 °C/min to 180 °C, retained for 2 min, after increased at a rate of 2 °C/min to 230 °C, retained for 2 min and finally increase at a rate of 5 °C/ min to 280 °C. The injection volume was 1μL in the splitless mode; n-hexane was used as solvent. The choice of this detector is carried out starting from several considerations: after hexane extraction, as proven by GC-MS, only chlorinated benzenes and PCB compounds are in the samples; concentration levels are not a critical factor since experiments are done in a controlled way and is possible to decide them, and the GC-FID measurements are supported and backed up by GC-MS in some experiments.

2.4. Sovtol-10 treatment for KPEG process

Powdered potassium hydroxide (KOH) was dissolved in PEG at 70°C before addition to the oil under treatment. The dehalogenation reaction was carried out in a 500 mL double jacket glass vessel. Contaminated oils were heated, and the KOH solution in PEG is added into the reaction vessel, to a temperature of 90 ± 1°C under continuous stirring with a turbine impeller at a rotation speed of 1000 rpm. With ultrasound, the reactor was directly immersed in the bath. An ultrasonic bath Model Brason 2200 operating at about 47 ± 6 kHz was used in this study. Different relations for KOH/PEG and KPEG/ oil were used.
All runs were operated in batch mode with five hours duration. Aliquots of 4 mL were collected at fixed times to analyze the PCBs content. Analyses were performed gas chromatographically after separating the glycolic phase (heavy) from organic phase (light).

The performance of this study was assessed in terms of the destruction and removal efficiency (DRE) of PCBs and the (DRE in percentage) was calculated by the equation (1):

\[
\text{DRE} = \frac{A_1 - A_2}{A_1} \times 100
\]

Where: \(A_1\) and \(A_2\) are the total area of peaks for mixture of the PCBs congeners before and after treatment.

3. Results and Discussion

3.1. Identification of the PCBs congeners in the Sovtol-10

The typical chromatogram of the Sovtol-10 is shown in Fig 1. In this chromatogram three Trichlorobenzene (1, 2, 4-TCBZ; 1, 2, 3-TCBZ and 1, 3, 5-TCBZ) and a variety of PCB congeners were identified. The total contents of tetra-, penta-, and hexachloro biphenyls were found to be about 11, more than 48, and about 29 %, respectively; minor amounts of trichloro- and heptachloro biphenyls were also detected. The PCBs congeners identified in the Sovtol-10 sample are shown in the Table 1.

![Chromatogram of Sovtol-10](image)

Fig. 1. Chromatogram of Sovtol-10 (the dotted line in the figure is the enlargement of the range 27-55 min)
Table 1. PCBs congeners identified in the sample

| Ballschmitter number | PCBs Congener                          | RRT        |
|----------------------|-----------------------------------------|------------|
|                      |                                        | PCB Mix 1 | Sovtol-10 |
| 18                   | 2, 2’,5-trichlorobiphenyl               | 0.803      | 0.802     |
| 31                   | 2,4’,5- trichlorobiphenyl              | 0.912      | 0.910     |
| 52                   | 2,2’,5,5’-tetrachlorobiphenyl         | 1.000      | 1.000     |
| 44                   | 2,2’,3,5-tetrachlorobiphenyl         | 1.053      | 1.052     |
| 101                  | 2,2’,4,5,5’-pentachlorobiphenyl      | 1.226      | 1.227     |
| 118                  | 2,3’,4,4’,5-pentachlorobiphenyl      | 1.371      | 1.373     |
| 153                  | 2,2’,4,4’,5,5’-hexachlorobiphenyl   | 1.414      | 1.414     |
| 138                  | 2,2’,3,4,4’,5’-hexachlorobiphenyl   | 1.463      | 1.464     |

3.2. Laboratory scale experiment

The effects of the KOH/PEG molar ratio, the KPEG/oil mass ratio and ultrasound on the PCBs destruction and removal efficiency (DRE) were determined using an experimental design 23. Reaction time was 120 min. The data obtained at 120 min are summarized in Table 2 where: X_1 is KOH/PEG ratio (mole) 1.2 and 1.5; X_2 is KPEG/oil ratio (wt.) 22 and 30; X_3 is the ultrasound and Y_1 and Y_2 destruction and removal efficiency DRE of PCBs.

Table 2. Overview of experimental design and overall DRE as a function of KOH/PEG, KPEG/oil ratio and ultrasound (reaction time 120 min)

| Runs | KOH/PEG | KPEG/oil | US  | Y_1  | Y_2  | Average DRE (%) | Standard deviation | RSD (n=2) (%) |
|------|---------|----------|-----|------|------|-----------------|--------------------|--------------|
| 1    | 1.5     | 30       | yes | 95.0 | 95.1 | 95.1            | 0.02               | 0.02         |
| 2    | 1.5     | 22       | yes | 93.6 | 93.3 | 93.4            | 0.19               | 0.20         |
| 3    | 1.2     | 30       | yes | 94.3 | 94.3 | 94.3            | 0.04               | 0.04         |
| 4    | 1.2     | 22       | yes | 93.4 | 93.3 | 93.3            | 0.09               | 0.10         |
| 5    | 1.5     | 30       | no  | 93.5 | 94.0 | 93.8            | 0.34               | 0.36         |
| 6    | 1.5     | 22       | no  | 92.7 | 93.0 | 92.9            | 0.22               | 0.24         |
| 7    | 1.2     | 30       | no  | 94.0 | 93.4 | 93.7            | 0.39               | 0.42         |
| 8    | 1.2     | 22       | no  | 92.3 | 92.7 | 92.5            | 0.25               | 0.27         |

A multiple linear regression model describes the relationship between DRE and the two independent variables at a specific reaction time. The equation of the fitted model (2) at 120 min of reaction is:

\[ \% \text{DRE} = 93.6 + 0.15X_1 + 0.59X_2 + 0.42X_3 + 0.057X_1X_2 + 0.07X_1X_3 + 0.05X_2X_3 + 0.11X_1X_2X_3 \]  

(2)

Where X_1 is KOH/PEG ratio, X_2 is KPEG/oil ratio and X_3 is ultrasound.
The results obtained for the factorial design 23 shows that tree independent variables considered significant influence the % DRE. The KPEG/oil ratio shows the most significant effect. The R-Squared statistic indicates that the model as fitted explains 95.874% of the variability in DRE.

Statistical comparison (using Statgraphic Program) between the means for the KOH/PEG molar ratio showed no significant difference at the 95.0% confidence level when comparing the KOH/PEG molar ratio 1.2:1 with KOH/PEG molar ratio 1.5:1.

The behaviours for four PCBs congener identified in the sample at different reaction time are summarized in the Table 3 as percentage of reduction (minus sign) or increment (plus sign) after 1 h and 2 h, respectively of treatment. The rest of the congeners identified disappear of the chromatogram at 60 minutes of the reaction time. The congeners taken as references were 18, 31, 52, 44, 101, 118, 153 and 138 according to the Ballschmitter number. The choice of these congeners was due to that these peaks are reproducible in the chromatogram and these were identified in the Sovtol-10 sample.

The results refer to Sovtol-10 treated at 90ºC with a rotation speed of 1000 rpm and KOH powdered. In all runs at one hour of the reaction a decrease of congener 18 is observed while the congeners 31, 52 and 44 shows an increment at the one and two hours of reaction. Therefore, the tendency for highly halogenated molecules to be converted into lighter compounds is demonstrated. The molecules PCBs of low number of chloro atoms were most difficult of dehologenate. The use of ultrasound leads to better efficiency compared to the results performed without ultrasound (Runs 1, 2, 3 and 4).

Table 3. Result of PCBs deshalogenation for the four congeners identified in Sovtol-10

| Congeners | 18 | 31 | 52 | 44 |
|-----------|----|----|----|----|
| 2, 2',5   |    |    |    |    |
| trichlorobiphenyl | -70.2 | -74.4 | 62.7 | -17.4 |
| trichlorobiphenyl | -63.0 | -66.1 | 96.7 | 10.6 |
| trichlorobiphenyl | -70.5 | -74.4 | 61.3 | -7.0 |
| trichlorobiphenyl | -70.5 | -73.2 | 61.3 | -7.0 |
| tetrachlorobiphenyl | -72.2 | -77.2 | 67.5 | -8.1 |
| tetrachlorobiphenyl | -59.8 | -63.7 | 115.6 | 18.9 |
| tetrachlorobiphenyl | -69.8 | -71.1 | 66.3 | -2.2 |
| tetrachlorobiphenyl | -53.8 | -61.0 | 155.9 | 26.3 |
| after 1 h | after 2h | after 1 h | after 2h | after 1 h | after 2h | after 1 h | after 2h |
| 72.4 | 30.8 | 145.3 | 87.7 | 183.9 | 37.2 | 104.6 | -49.4 |
| 72.4 | 30.8 | 145.3 | 87.7 | 183.9 | 37.2 | 104.6 | -49.4 |

3.3 Preliminary kinetic study of the removal of PCBs from Sovtol-10

With the best experimental condition obtained in the previous experiment a new experiment at higher reaction times was realized. The % DRE was determinate each one hour; the results are shows in the Fig 2.
As shown in Fig 2 there is an increase in DRE to 70% in the first 30 minutes of the reaction. After this time it increases more slowly, attaining values of about 99.6% at the five hours of reaction. However, at time between three and five hours it increases only of 0.6%.

During the dehalogenation kinetic study, the DRE of mixtures TCB and PCBs were determined every 10, 20, 30, 60, 120, 180, 240 and 300 minutes of reaction. The dehalogenation kinetics of TCB and PCBs were showed in Fig 3.

It was found that the DRE of Sovtol-10 increases exponentially with reaction time in the first 30 min. The TCB mixture disappears of the chromatogram at 30 min of reaction time. In PCBs case the DRE increases rapidly with dehalogenation time until 60 minutes, then this increase becomes slower. Regression on the experiment data delivered the first order reaction rate coefficient ($k_1$) are $1.08 \times 10^{-1}$ ($R^2 = 99.09, n = 5$) and $1.48 \times 10^{-2}$ ($R^2 = 98.74, n = 5$) at $90 \pm 1 ^\circ C$ for TCB and PCBs mixture respectively. The intercept of the adjusted line (0.9117) indicates that there are PCBs congeners to end of the reaction time studied.

The kinetic of PCBs deshalogenation to $90 \pm 1 ^\circ C$ involve a PCBs mixture composed fundamentally of several tetra-, penta- and hexa-PCBs that not shows the same kinetic behaviour. The results obtained, show that reaction rate is not the same for all PCBs homologues (Fig 4). The highly chlorinated biphenyls were more quickly eliminated than the slightly chlorinated ones which tend to increase at the first hour of reaction. In the chromatogram appear di- and tri- PCBs congeners that not are present in the Sovtol-10 sample. However, after one hour of treatment a decrease for all congeners was observed by indicating the effectiveness of the method.
In the sample remain the 52 and 44 PCBs congeners and appear di- and tri-chlorinated congeners, which were not detected in the original sample. These congeners are not reported like toxics for the environment by the WHO. Therefore, much additional research of the mechanism of this reaction is necessary.

3.4 Scale-up of the KPEG process

Starting from the results obtained at laboratory level is carried out the design of an experimental plant-pilot of treatment that allow to process the polluted oil of Villa Clara, Cuba and besides, to continue the experimental work in order to present a proposal of an industrial plant. The experimental plant is calculated for a daily capacity of treatment of 21 kg of oil with PCBs, in two batches a day according to the outline that is shown in the Fig 5.
deshalogenation were selected two stirred double jacket reactors, to assure the temperature control (70 °C in the first reactor and 90 °C in the second reactor). Mixing is achieved by a turbine-impeller at 1000 rpm.

The scale-up of these reactors is assumed as mixers, since the reaction time is eight times larger than mixing time. [15]

The following conditions are assumed in the scale-up:
- Constant stirrer speed, because the mixture of the reagents is a fundamental element in the process. [16]
- There is a geometric similarity between the model and prototype diameter according to the equation 3. [17]

\[
\frac{D_{\text{reactor}}'}{D_{\text{reactor}}} = \frac{d_{\text{impeller}}'}{d_{\text{impeller}}} = \left(\frac{V_{\text{total}}'}{V_{\text{total}}}\right)^{\frac{1}{3}}
\]

The Table 4 shows the model dates and in Table 5 appears the obtained results for the prototypes. Both reactors should be built of stainless steel 430 AISI (American Iron & Steel Institute).

Table 4. Date of stirrer tank (model)

| Total volume (Vt) | 0.5 L |
|-------------------|-------|
| Effective volume (v) | 0.4 L |
| Reactor diameter (Dr) | 0.075 m |
| Impeller diameter (D = 0.8 Dr) | 0.06 m |
| Stirrer speed (N) | 1000 rpm |
| Height of the tank (H) | 0.13 m |
| Filling height of the tank (h) | 0.11 m |
| Reaction temperature (Tr) | 70 y 90 °C |

Table 5. Results obtained for prototypes

|                      | Prototype 1 | Prototype 2 |
|----------------------|-------------|-------------|
| Reactor diameter (m) | 0.58        | 0.58        |
| Impeller diameter (m) | 0.47       | 0.47        |
| Total volume (l)     | 250         | 250         |
| Reaction temperature (°C) | 70        | 90          |
| Stirrer speed (rpm)  | 1000        | 1000        |
| Stirrer power (kW)   | 117.49      | 103.75      |
| Vapour mass (kg)     | 8.23        | 4.10        |
| Heat transfer area (m²) | 0.892    | 0.578       |
3.4.1 Design of the heat exchanger

For the design of the heat exchange was used the methodology proposal for Kern. [18] Considering a stainless steel double pipe heat exchanger with an inside diameter of 3.17 $10^{-2}$ m and an outside diameter of 5.08 $10^{-2}$ m, and besides a hairpin length of 2.5 m, five hairpins are necessary.

3.4.2 Design of the auxiliary equipment

The results for the design of storage tanks for PEG 400 and PCBs oil and the separator appear in the Table 6. All these equipment will be built of stainless steel.

Table 6. Design of auxiliary equipment (D= Diameter and H= Height)

| Equipment                  | Capacity       | Dimensions         |
|----------------------------|----------------|--------------------|
| Storage tank for PEG 400   | 0.178 m$^3$    | D=0.5 m; H=0.64 m |
| Storage tank for PCBs oil  | 0.00618 m$^3$  | D=0.16 m; H=0.367 m|
| Separator                  | 0.1958 m$^3$   | D=0.5 m; H=0.731 m |
| Nutsche Filter             | 0.012 m$^3$    | D=0.35 m; H=0.124 m|
| Storage tank for residues  | 0.5 m$^3$      | D=0.8 m; H=1.10 m  |

A solid screw feeder is proposal to add KOH powdered into reactor. The design was made using the norm UNE 58-244-8819 and to estimate the power the methodology proposal for Ulrich is used. The conveyer diameter is fixed in 0.2 m and the conveyer length in 0.8 m, with these value is calculated the screw speed, it is 15.6 rph and the input power may be 0.083 kW.

In this process are used two semi-open impeller centrifugal pumps, one is to discharge the KPEG formed in the firth reactor into the second one, and the other pump is used to move the reaction products to the heat exchanger. The capacity for both pumps is 0.003 m$^3$/min and 0.25 kW of power are necessary. The proposed flow diagram for the treatment of Sovtol-10 using the KPEG process is showed in Fig 6.
Fig. 6. Flowchart of experimental pilot-plant for chemical decontamination of Sovtol-10, 1, screw feeder; 2, storage tank for Sovtol-10; 3, storage tank for polyethylene glycol 400; 4, chemical reactor for the formation of KPEG; 5, chemical reactor for decontamination of oil; 6-7, pumps; 8, heat exchanger; 9, separator; 10, Nutsche filter and 11, storage tank for aryl poly(ethylene glycol)

4. Conclusions

In the sample of Sovtol-10 were identified several tetra-, penta- and hexa-PCBs that include the congeners 18, 31, 52, 44, 101, 118, 153 and 138.

PCBs dehalogenation in Sovtol-10 involves all congeners with high efficiency. In particular, congeners of high molecular mass disappear from the chromatogram in all runs. The best results were attained under the following conditions: KOH/PEG molar ratio 1.5/1, KPEG/oil weight ratio of 30 and with ultrasound to two hour of reaction. The removal of PCBs is strongly affected by KPEG/oil ratio.

Experimental runs at laboratory scale showed that the kinetics of the removal reaction was first-order for TCB mixture and first-order for PCBs mixture.

The KOH/PEG molar ratio 1.2/1 and KPEG/oil weight ratio of 22 was selected for the plant-pilot design because there are no significant difference at the 95.0% confidence level when the different KOH/PEG molars ratios are compared. Average DRE of Sovtol-10, was 99% at three hours of reaction.

A plant pilot experimental for the treatment of Sovtol-10 using the KPEG process was designed. The chemical reagents PEG and KOH used are common and fairly inexpensive and the operation of the reactors system is simple and safe, requiring no special precautions.

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