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ABSTRACT

A technically & economically sound technology for water detritiation is mandatory for the future of fusion. This technology is expected to be based on water electrolysis and Liquid Phase Catalytic Exchange (LPCE). LPCE requires an efficient hydrophobic catalyst. SCK•CEN invented and developed such a catalyst in the past, which is prepared by depositing platinum on an activated charcoal carrier and mixing it with polytetrafluorethylene as a hydrophobic material. In combination with an appropriate wetttable packing, different batches of this catalyst performed very well during years of extensive testing, allowing us to develop the ELEX process for water detritiation at inland reprocessing plants. Recently we succeeded in reproducing this catalyst and preparing a slightly different but clearly ameliorated type. By extrapolation these new results would allow us to obtain, at 40°C and under typical but conservative operating conditions, a decontamination factor of 10000 with a column of less than 3 meters long. Such performances would make this catalyst an excellent candidate for application at JET or ITER. To confirm the performances of our improved catalyst for a longer period of time and in a longer column, we are now starting experiments in a newly built installation and we are collaborating with ICSI, Romania.

1. INTRODUCTION

Fusion needs water detritiation. The production of fusion energy is expected to involve the use of huge amounts of tritium, of the order of $1.5E19 \text{ Bq GWth}^{-1} \text{ a}^{-1}$. Fusion reactors have to handle 50000 times more tritium than present Light Water Reactors (LWRs). Therefore their management of tritium losses should not be based on discharging and diluting, as is being done for LWRs, but on containment. Operation and dismantling of test installations and future commercial fusion reactors appear impossible without the production of tritiated waste. Most of this waste exists as or can easily be transformed into tritiated water. Segregation should limit the volume of accumulated tritium containing water. A further volume reduction can be obtained by water detritiation, which splits up the tritium contaminated water in a large nearly tritium free fraction that can be discharged and a small fraction with nearly all the tritium. The latter is then further treated for conditioning and storage, or for tritium recovery.

Contrary to tritium removal from closed systems such as Heavy Water Reactors (HWRs), detritiation of water for environmental release, such as for fusion applications, is expected to need high decontamination factors [1]. Therefore it makes favorably use of the isotopic exchange of tritium between hydrogen gas and liquid water, the large isotope effect of which is easily multiplied in a countercurrent trickle bed reactor. The equilibrium constant for the tritium exchange reaction is close to 6 at 40°C and it decreases with increasing temperature. The equilibrium isotope effect favors concentration of tritium in the liquid phase, relative to the hydrogen gas. Because Liquid Phase Catalytic Exchange (LPCE) uses liquid water, the application of a countercurrent and thus the multiplication of the elementary separation steps are nearly as easy as for rectification.

For water detritiation, most recent designs use a so-called Combined Electrolysis – Catalytic Exchange (CECE) process, although also dual temperature applications might be envisaged. A CECE

installation comprises essentially an electrolysis cell for the production of (tritiated) hydrogen by dissociation of (tritiated) water and a countercurrent packed-bed reactor (LPCE column) for tritium transfer from hydrogen gas to liquid water. As the liquid water trickles down the LPCE column, it becomes more and more enriched in tritium. The hydrogen gas, which is already depleted in tritium relative to the electrolyte solution from which it evolved, becomes more and more depleted in tritium when it flows from bottom to top. The tritium contaminated water is fed into the LPCE column at the point where its tritium content corresponds to the local tritium content of the liquid phase. Tritium free water is added at the top of the column. The tritium depleted hydrogen stream (cfr decontamination factor) at the top of the column is vented and at steady state a small fraction of tritiated enriched water (cfr enrichment factor or volume reduction factor) can be removed as condensate from the electrolysis cell for immobilization and storage. For the recovery of pure tritium by further processing, one removes a small fraction of the electrolytically produced hydrogen gas before it enters the LPCE column. From the above description of the CECE process it shall be clear that the ratio of the molar hydrogen and water flow rates in the enrichment part of the column (below the feed point) is normally close to one, because (nearly) all water is dissociated in the electrolysis cell. In the stripping part of the column (above the feed point), this ratio should be as high as possible for economic reasons. But it cannot be higher than the equilibrium constant of the exchange reaction at the operating temperature of the column. Otherwise there is not enough water to remove all tritium from the hydrogen, even at an infinitely high exchange rate in an infinitely high column.

LPCE and thus also water detritiation by CECE require an efficient catalyst that should furthermore be hydrophobic because of the low solubility and hence slow diffusion of hydrogen in the water layer that covers normal catalysts. Since more than 30 years, several groups have been developing such catalysts [2]. Encouraged by early Canadian ideas [3], SCK•CEN, the Belgian nuclear research center, was the first but one to invent and develop a LPCE catalyst, which was based on fuel cell technology [4]. In combination with an appropriate wettable packing, different batches of this catalyst performed very well during thousands of hours and in 0.015 to 0.1m diameter LPCE columns with different heights. This work and the support of Europe and FZK, Germany, allowed us to develop the ELEX process for water detritiation at inland reprocessing plants, which was in fact the Belgian equivalent of the CECE process [5]. But when inland reprocessing was abandoned in Belgium and in Europe, the project was shutdown for more than a decade. A few years ago, and with the support of the European Fusion Development Agreement (EFDA), we restarted some work in order to reproduce and improve the historical performances of the SCK•CEN catalyst and its combination with water wettable inerts. As a result we hope to demonstrate that a relatively small LPCE column (with an electrolysis cell) should be sufficient for most future water detritiation applications.

2. UNDERLYING PRINCIPLES

The SCK•CEN catalyst is prepared by depositing platinum on an activated charcoal carrier and mixing it with PolyTetraFluorEthylene (PTFE) as hydrophobic material. The composition and shape of the original SCK•CEN catalyst and its detailed preparation have been described elsewhere [4].

At the moment SCK•CEN relies heavily on its agreements with specialised firms for the preparation of catalyst batches. For LPCE application we filled a countercurrent trickle bed reactor with a uniform mixture of catalyst particles and water wettable, high surface area inerts of the same size, e.g. etched stainless steel spirals.

In an operating LPCE column the porous hydrophobic catalyst particles are filled with stagnant hydrogen gas and water vapour but they are not accessible to liquid water. The upwards flowing hydrogen gas is saturated with water vapour at the operating temperature and it fills the open space of the reactor. The liquid water flows down as a film which covers the wettable external surface of the inerts. The exchange of tritium between hydrogen gas and liquid water requires then several steps:

1. Evaporation of water at the surface of the downwards flowing film that covers the inerts.
2. Transport of water vapour to the catalyst particle through the upwards flowing gas phase.
3. Transport of tritiated hydrogen to the catalyst particle.
4. Diffusion of the reactants (tritiated hydrogen and water vapour) into the catalyst particle.
5. Chemisorption of the reactants, tritium exchange reaction and desorption of the reaction products (hydrogen and tritiated water vapour).
6. Diffusion of the reaction products out of the catalyst particle.
7. Transport of tritiated water vapour to the liquid surface through the upwards flowing gas phase.
8. Condensation of tritiated water vapour at the surface of the downwards flowing liquid film that covers the inerts.

Each of the cited steps might determine or influence the overall tritium exchange rate and thus the detritiation performance of the column. Therefore not only the catalyst particles (accessible Pt area, hydrophobicity, diffusion resistances) but also the inerts (surface area, wettability) and their mixing with the catalyst particles (ratio, uniformity) are important. Furthermore the performance is largely influenced by the operating conditions of the counter current trickle bed reactor, i.e. the temperature and the pressure, the volumetric gas flow rate and the ratio of the molar flow rates of hydrogen and water.

To test the influence of the catalyst itself, we tried to keep the other parameters constant over the different tests. Their values were chosen on the basis of representativeness for the later application, physical constraints, past experience and common sense. Figure 1 illustrates the principle of our testing of a combination of LPCE catalysts particles and wettable inerts. The tests are carried out at a given temperature (mostly 40 or 60°C) and pressure (normally atmospheric) in a counter current trickle bed reactor which is filled over a height Z with the to-be-tested catalyst particles and etched stainless steel spirals of about the same size, uniformly mixed in a volumetric ratio of e.g. 1:2. The tests are carried out a specific molar flow rate G (typically $10 \text{ mol s}^{-1} \text{ m}^{-2}$) of hydrogen and a ratio G/L (typically 3, lower is easier, higher is more difficult) between the specific molar flow rates of hydrogen and water. There are several constraints for the choice of these working conditions. A lower temperature is better because of the higher equilibrium isotope effect and the limited presence of water vapour (cocurrent with hydrogen). On the other hand reaction rate constants tend to decrease at

lower temperature and a relatively too low water vapour concentration could further decrease the rate of step 5 above, unless the overall pressure is reduced. A higher specific molar gas flow rate G should increase the overall mass transfer and thus the exchange rate, but eventually G is limited by flooding. If decontamination of hydrogen is aimed at, the ratio G/L should be lower than the value of the exchange equilibrium constant at the operational temperature. But decreasing G/L for the stripping column increases the cost of detritiation by the CECE process. Finally at atmospheric pressure and at a G/L ratio that is not smaller than one, the specific molar liquid flow rate L is below the minimum wetting rate of normal column internals. Therefore the need to use very wettable (etched) high performance inerts and our preference to flood the column before a test or a series of tests. The tritium concentrations in the hydrogen (y) and in the water (x) are measured at the top of the LPCE column, where the water (mostly tritium free) is fed, (x_{in} and y_{out}), and at the bottom of the column, where the (tritiated) hydrogen enters (y_{in} and x_{out}).

At steady state the most important indicator for the performances of a given LPCE column under specified operating conditions is the tritium decontamination factor (DF), i.e. y_{in}/y_{out} or the ratio between the tritium concentrations in the hydrogen gas before (at the bottom of) and after (at the top of) the column. As however identical columns and conditions cannot always be used, different authors have applied different models that should allow obtaining somewhat more generalized modelling results, including ours, ask for experimental verification.

Consider a counter-current trickle bed reactor with a height Z and a cross section A , as represented in Fig.1, in which the concentration of HT decreases from y_{in} at the bottom to y_{out} at the top [6]. At a height z in the column, the overall exchange rate is given by:

$$Gdy = k (y_e - y) dz$$

Integration over the whole column leads to:

$$\begin{aligned} Z &= \frac{G}{k} \int_0^Z \frac{dy}{y_e - y} \\ &= \frac{G}{k} \frac{(y_{out} - y_{in})}{(y_e - y)_{out} - (y_e - y)_{in}} \ln \frac{(y_e - y)_{out}}{(y_e - y)_{in}} \end{aligned}$$

where

k = the overall exchange rate constant based on the gas phase and

y = the tritium concentration of the hydrogen at a certain height in the column

y_e = the tritium concentration that the hydrogen would have at that height if its tritium concentration would be in equilibrium with the tritium concentration x of the water at that same height in the column:

$$y_e = \frac{x}{K} \quad \text{with } K = \text{the equilibrium constant of the reaction: } \text{HT}_{(g)} + \text{H}_2\text{O}_{(l)} \rightleftharpoons \text{H}_{2(g)} + \text{HTO}_{(l)}$$

By definition:

$$H_{OG} = \frac{G}{k} \quad \text{and} \quad N_{OG} \int_0^{\approx} \frac{dy}{y_e - y}$$

Hence:

$$Z = H_{OG} \times N_{OG}$$

H_{OG} is the height of a transfer unit based on the gas phase. It measures the separation effectiveness. It should be as small as possible.

N_{OG} is the number of transfer units based on the gas phase. It measures the difficulty of the separation.

When testing with tritiated hydrogen and/or tritiated water in a LPCE column with a known height (Z) and under specified conditions, this model allows to calculate, from the tritium concentrations at the bottom (y_{in}) and the top (y_{out}) of the column, the number of transfer units (N_{OG}) and thus also the height of a transfer unit (H_{OG}) under these conditions.

In a later design phase, the same model shall allow the calculation of the number of transfer units (N_{OG}) that are required for a to-be-realised separation, and thus also the required height (Z) of the LPCE column under operating conditions for which the height of a transfer unit (H_{OG}) is known.

This simplified model does not take into account the water vapour inside the column, and the water vapour entering the column both on top (coming from the condenser) and at the bottom (coming from the saturator). The influence of the vapour is assumed to be limited surely when working at lower temperatures (40°C) and moderate G/L.

An alternative variable which is sometimes used to measure the separation effectiveness is the height of a theoretical plate (HETP). A relation between H_{OG} and HETP can be described as follows [7]:

$$HETP = H_{OG} \frac{\ln \left(\frac{K}{G/L} \right)}{1 - \frac{G/L}{K}}$$

3. EXPERIMENTAL SETUP AND PROCEDURE

For measuring and demonstrating the performances of LPCE catalysts, SCK•CEN has built several different test installations, already more than 25 years ago and up to the present. Each installation comprised a thermostatically controlled countercurrent trickle bed column that contains the to-be-tested mixture of hydrophobic catalyst particles and wettable inerts, with a feed of tritiated hydrogen and a feed of (mostly) tritium free water. Recently we have been using glass columns with an internal diameter of 0.02m. In the past we used glass, plexiglass and stainless steel columns of 0.015 to 0.1m diameter and 0.4 to 3.4m height. To avoid flooding at the bottom of the column we use a downwards facing conical support for the smaller columns. With larger columns special bottom pieces have been used, as well as liquid distributors at the top of the column. The catalyst packing mixture is inserted between two layers of wettable inerts, at the moment each 0.2m high.

The dry and mass flow controlled feed tritiated hydrogen has been produced by blending bottled tritiated hydrogen and pure hydrogen or by electrolytic dissociation of (tritiated) water. More recently

it has been prepared by isotopic exchange between pure hydrogen and tritiated water, using our own hydrophobic catalyst. First we passed the hydrogen through a large bubble bed reactor that contained the catalyst immersed in tritiated water. Now the use of a second and overdimensioned thermostatically controlled countercurrent trickle bed reactor that is fed with water of a known tritium concentration at a G/L ratio below the value of K (at the operating temperature), provides us with hydrogen of a constant and known tritium concentration. If necessary the tritiated hydrogen feed is dried by condensation and adsorption of its water content. It is then sampled and its mass flow rate is measured before it is re-saturated at the bottom of the test trickle bed reactor. Also after the test trickle bed reactor the water is removed from the (decontaminated) hydrogen and returned at the top of the test column. A calibrated pump adds tritium free water at the top of the column. The (average) liquid flow rate is furthermore checked by weighing the incoming and outgoing water.

The tritium concentration in the dry hydrogen gas before (y_{in}) and after (y_{out}) the test trickle bed reactor is measured by means of calibrated proportional counters. Since we have been using a trickle bed column for the preparation of the tritiated hydrogen feed, we furthermore calculate y_{out} from K and the measured tritium content of the tritiated water feed. The tritium concentration of the water after the test column (x_{out}) is measured using a calibrated liquid scintillation counter.

From the known specific flow rates of hydrogen (G) and water (L) that pass through the test column and from the measured tritium concentrations y_{in} , y_{out} and x_{out} at steady state, e.g. after one night of stabilization, ($x_{in} = 0$) we check the overall tritium balance. If the tritium balance proves to be in equilibrium, i.e. when equal amounts of tritium enter and leave the test trickle bed reactor, the decontamination factor y_{in}/y_{out} is a measure for the performances of the LPCE column and its filling of catalyst particles and wettable inerts, at the operating conditions that were used. The above described model allows us then to obtain the experimental value of NOG, for the LPCE test column and thus also the height of an overall exchange transfer unit based on the gas phase, HOG (or HETP, height of a theoretical plate) and the overall exchange rate constant k.

4. RESULTS AND DISCUSSION

Many tests have been carried out at SCK•CEN on different test batches that have successively been prepared. Here we shall refer to results that were obtained after 1980 or even after 2000 when we restarted preparing and testing catalyst batches after more than a decade without comparable activities at SCK•CEN. As explained in II, most tests were realized at 40 or 60°C and at atmospheric pressure. G, the specific hydrogen flow rate through the LPCE column was usually about $10 \text{ mol s}^{-1} \text{ m}^{-2}$, and G/L, the ratio of hydrogen and water molar flow rates was either around 1 (cfr CECE, enrichment) or around 4 (cfr CECE, stripping).

The to-be-tested cylindrical particles were prepared by depositing platinum on an activated charcoal carrier and mixing it with polytetrafluorethylene (PTFE). The mixture is pressed into a cake and then rolled into sheets. Finally catalyst particles with a height of about 2mm and a diameter of about 1.9mm were punched from the sheets. For most steps in this procedure we could rely on specialized commercial firms. The hydrophobic particles were uniformly mixed with etched commercially available

stainless steel spirals of about the same size in a volumetric ratio of 1:2 or in some cases 1:3.

Between 1980 and 1985, several series of experiments with catalyst particles composed of 80 wt% PTFE, 19 wt% C and 1 wt% Pt and mixed with wettable inerts, were carried out in 0.02 and 0.1m diameter stainless steel columns and in a 0.03 m plexiglass column. The height of the catalytic bed varied between 0.7 and 2.1m for the 0.02m diameter column, 0.7 to 1.15m for the 0.03m plexiglass column and 0.9 to 3.5m for the 0.1m diameter stainless steel column. The length of the tests varied from about 20h inclusive stabilization over night to more than 5000h. Except for abnormal situations, such as an incidental interruption of the feed of tritium free water, or specific problems such as outgoing tritium in hydrogen concentrations below the detection limit of the proportional counter, nearly all tests for all our catalyst batches gave overall exchange rate constants between 40 and 120 mol s⁻¹ m⁻³. At a specific hydrogen flow rate G of 10 mol s⁻¹ m⁻², HOG, the height of an overall exchange transfer unit based on the gas phase, was thus always smaller than 0.25 m but larger than 0.08m. The corresponding HETP values vary depending on temperature and G/L ratio but they were situated between about 0.1 and 0.5m. Of course the obtained decontamination factors varied with the length of the LPCE column and other parameters such as G and the G/L ratio. In the 0.03 m diameter plexiglass column with 1.15m of the catalytic mixture at 40°C and at G/L = 1 we measured decontamination factors up to more than 5000. In the 0.1 m diameter stainless steel column, a DF of up to 3600 was obtained with 3.5 m of the catalytic mixture but at G/L = 3.7 and G = 12 mol s⁻¹ m⁻².

Since a few years we have been trying to reproduce and improve the SCK•CEN LPCE catalyst and its historical performances. A first new batch was a reproduction of the “reference” catalyst we produced 20 years ago. Preliminary tests at SCK•CEN resulted in transfer units, HOG, between 0.2 and 0.25m at 40°C and at a specific hydrogen flow rate of 10 mol s⁻¹ m⁻². Testing was also done at the D. Mendeleev University in Moscow with the assistance of JET, and at the Tritium Laboratory of the Forschungszentrum Karlsruhe [8,9]. These results confirmed our maintained ability to prepare the catalyst. In 2002 a few new batches were prepared. A reference batch was made according to the original catalyst specifications, but we also prepared batches with only 70 wt% PTFE instead of 80 wt%, with 2 wt% Pt instead of 1 wt%, with two different versions of a different charcoal type support (advised by the supplier) and with a high surface graphite support. All catalysts were tested several times at 40 and 60°C, G = 10 mol s⁻¹ m⁻² and G/L between 3.4 and 3.55. Because of the comparative character of these experiments only short tests (over night stabilization) and short beds (0.5m) were used. The reference catalyst yielded an overall exchange rate constant (k), based on the gas phase, of 54 mol s⁻¹ m⁻³ at 40°C (H_{OG} = 20cm) and 78 mol s⁻¹ m⁻³ 14cm). Although the 70 wt% PTFE catalyst contained relatively more Pt than the reference, it performed slightly worse. The high graphite catalyst was nearly as good as the reference and the 2 wt% Pt catalyst performed about 20% better. The catalysts with the different charcoal support yielded the best results, k values of 93 and 123 mol s⁻¹ m⁻³ at 40°C and 127 and 139 mol s⁻¹ m⁻³ at 60°C. For the best catalyst HOG, the height of a transfer unit, was 9 cm at 40°C and 8 cm at 60°C, corresponding to a HETP of 11.4 and 9.6cm respectively. Although the height of a transfer unit at 60°C is lower than the one at 40°C, one should not forget that at a higher temperature more transfer units are required to obtain the same decontamination [10].

From II, the following equation can be derived to calculate N_{OG} , the (minimum) number of transfer units required to obtain a certain decontamination factor DF at a given G/L ratio and at a given temperature:

$$N_{OG} = \frac{1}{1 - \frac{G/L}{K}} \ln \left(DF - \frac{G/L}{K} (DF-1) \right)$$

The results of our earlier catalyst preparation and testing and especially those of our recent work are so good that, contrary to presently proposed designs, very short columns (less than 3m) could be sufficient to obtain very high decontamination factors (10000), even at a high molar flow ratio G/L. As furthermore also relatively high hydrogen flow rates ($10 \text{ mol s}^{-1} \text{ m}^{-2}$ or more) are applicable, also the column diameter and thus the amount of LPCE catalyst that is needed for water detritiation by the CECE process can be rather small.

As our most recent and best results were obtained in an only 0.5m high column, extrapolation to very high decontamination factors should also experimentally be verified and studied. Furthermore the efficiency of the catalytic mixture and the realization of very high decontamination factors should be tested and verified over longer periods. Therefore we are now preparing LPCE experiments with 2m of a uniform mixture of etched stainless steel spirals and the to-be-tested better catalyst particles. To assure a constant tritium concentration, an additional trickle bed reactor is used for the preparation of the tritiated hydrogen feed, as explained in III. In parallel endurance tests for the SCK•CEN mixture are under way at ICSI, Romania in collaboration with SCK•CEN.

Finally it should be mentioned that all LPCE experiments that up to now have been carried out at SCK•EN aimed at the detritiation of light water and did not take into account the possible presence of appreciable amounts of deuterium. At SCK•CEN the impact of H, D and T species present in the LPCE column shall be determined next year.

CONCLUSIONS

A technically and economically sound technology for water detritiation is an environmental prerequisite for the further development and the future application of fusion energy. This water detritiation shall at least partly be based on the large isotope effect during the exchange of tritium between hydrogen gas and liquid water at relatively low temperature. Such Liquid Phase Catalytic Exchange (LPCE) requires an efficient catalyst that is not poisoned by liquid water.

In the past SCK•CEN invented and developed a LPCE catalyst, which is prepared by depositing platinum on an activated charcoal carrier and mixing it with polytetrafluorethylene as hydrophobic material. Together with the catalyst itself a testing procedure evolved that has been applied since more than 25 years ago on several batches of the SCK•CEN catalyst mixed with wettable inerts. Although our catalyst was tested in different LPCE columns of various diameter and lengths, and during shorter and longer time periods, overall exchange rate constants (at 40 or 60°C and at a specific hydrogen flow rate of $10 \text{ mol s}^{-1} \text{ m}^{-2}$) of the order of $50 \text{ mol s}^{-1} \text{ m}^{-3}$ or larger were consistently obtained during a decade.

SCK•CEN's R&D on water detritiation has been suspended for a long period but recently we succeeded in reproducing and even ameliorating our catalyst and its historical performances. The present results, overall exchange rate constants of the order of $100 \text{ mol s}^{-1} \text{ m}^{-3}$, even at 40°C and at a gas to liquid molar flow ratio of 3.5, are so good that a 3 m LPCE columns would be sufficient to obtain decontamination factors of the order of 10000 under anticipated economically favorable working conditions. In the near future we hope to confirm these results during longer tests in a longer LPCE column and in collaboration with other European partners.

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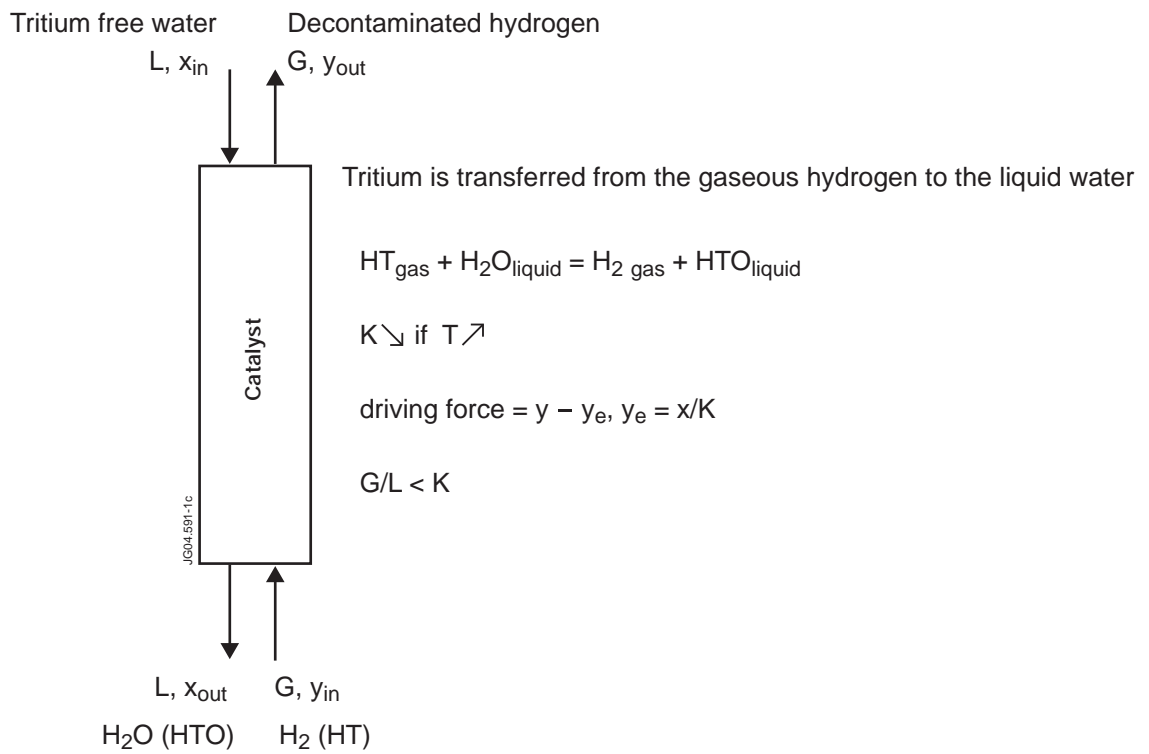


Figure 1: LPCE uses a catalyst in a counter-current trickle bed reactor