



Effect of pH of Medium and Initial Salt Quantity on Release Behaviour of Potassium Chloride from Leached Poly(vinyl alcohol)/Glutaraldehyde

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The purpose of present work is to study the release kinetics of KCl from leached poly(vinyl alcohol)/glutaraldehyde (PVA/GA) hydrogels into demineralized water under various pH conditions. The PVA/GA hydrogels, which was prepared by mixing method, were leached several times using demineralized water. The physico-chemical properties of the hydrogels were investigated using FT-IR and SEM techniques. The release kinetics of KCl from the hydrogel into the demineralized water at various pH conditions and initial quantity of KCl inside the hydrogel were studied using conductometric method. It was found that the trapped residual inside the hydrogel matrix can be well removed by repeated leaching. The pH of medium affects the rate of release and the amount of KCl at equilibrium conditions. The kinetics of KCl desorption into the media follows pseudo first-order kinetics, which indicates that the release kinetics is controlled by the initial amount of KCl in the PVA/GA matrix.

Keywords: Conductometry, Leaching, PVA/GA hydrogel, Potassium chloride, Release.

INTRODUCTION

Because of its vital role of potassium in many aspects of life, especially for living systems, has become the focus of study in many scientific fields [1]. In agriculture, especially related to fertilizers, potassium has been intensively studied both for its role as a living system substance and to control its presence as the source of nutrient intake [2,3]. The presence of sufficient potassium can increase nitrogen use efficiency (NUE) [4]. This happens because potassium can affect the activity of enzymes involved in C and N metabolism, nitrate transport, morphology and root activity. To maintain the availability of nutrients (including potassium) around the roots, fertilizer technology has been developed using the slow-release fertilizer (SRF) or controlled-release fertilizer (CRF) approach [5-9]. One of the fertilizers packaging forms in the SRF or CRF approach is to utilize hydrogel, a 3D polymeric material that can be obtained through synthesis and/or from natural polymers that have the ability to absorb water and retain it significantly [10,11]. Among the hydrogels that have been commonly studied for this purpose are hydrogels based on polyvinyl alcohol (PVA) with glutaraldehyde (GA) crosslinkers, which

were developed not only for agricultural purposes but also for the medical field [12].

In agricultural applications, in addition to being important to understand its physico-chemical properties, it is also important to understand the agrochemical performance of the hydrogel under consideration. One of the agrochemical performances of hydrogel-encased fertilizers is the kinetics of nutrient desorption from the gel into the environment. There are many factors that can affect the characteristics of the desorption or release of nutrients from the hydrogel to the environment, including the physico-chemical properties of the gel itself and its environment condition. The intrinsic properties of gels, such as pore diameter, cavity area, cavity surface charge, functional groups and hydrophilicity are parameters that generally of interest. The penetrability of the gel by the solvent is one of the determining factors of the desorption rate. Gels with large cavities, where the charges and functional groups of cavities increase the voids in the gel, making it easier for solvent to penetrate into the gel [13-16].

Environmental conditions such as pH, ionic strength, polarity, types of solvent and temperature are often become parameters that need to be considered in desorption studies

[15-17]. Condition of medium can affect the physico-chemical state of the gel, producing a thermodynamic state in the system for the particle of solute to transfer between phases both chemically and physically. It has been also reported that the desorption of potassium from both biotite and K-feldspar depended on the nature of the organic acids used and their composition. Compared to K-feldspar, biotite in general desorb more potassium. It was also found that the addition of oxalic acid tends to increase K-desorption ability of biotite and K-feldspar in comparison to citric acid [18].

The release of KCl from the hydrogel made from PVA and borate indicated that a medium within a solvent with high polarity was preferred, as indicated by a decrease in the release constant with decreasing solvent polarity [17]. Besides, the nature of solute itself is a variable of interest in the performance study of solid fertilizer. It has been reported that the release rates of various materials in organic acids were $(Al, Fe, Mg) > K > Si$ [18]. In addition, the leaching levels of K^+ , Ca^{2+} and Mg^{2+} were greater than that of N and P from fertilizer tablets and soil tablets [19].

The use of the conductometric method in studying the kinetics of release from gel into media as well as permease between membrane-separated liquid has ever been carried out. Practically, this method is favourable since it may be utilized for assessment frequently, although other ions may interfere with system conductivity. Saeed & Abdeen [17] used this method to study KCl release from hydrogels made from PVA and borate. Hendrawan *et al.* [20] used this method to study the permease of KCl from its solution into deionized water through PVA/glutaraldehyde/*Premna oblongifolia* Merr. membrane. Cancellier *et al.* [21] examined the conductivity method for assessing nutrient release from controlled-release fertilizers and found that it agreed well with other analytical techniques.

The investigation of the kinetics of KCl release was conducted on hydrogel media under different acidity conditions and varying quantities of KCl. Consequently, the cleanliness of the hydrogel has emerged as a significant issue of consideration. In this work, the effect of pH of media and the initial quantity of KCl inside the hydrogel to the release kinetics of KCl using conductometric method as well as the leaching of residual ions from inside the gel were studied, where the results is expected to be beneficial in the development of the controlled-release fertilizer (CRF) materials.

EXPERIMENTAL

Materials used include poly(vinyl alcohol) (PVA) p.a. with molecular mass 60,000 g/mol (Merck, Germany), glutaraldehyde (GA) 25% with $\rho = 1.055\text{--}1.065$ g/mL (Merck, Germany), sulfuric acid 97% with $\rho = 1.474\text{--}1.480$ g/mL (Merck, Germany), glacial acetic acid p.a. with $\rho = 1.05$ kg/L (Merck, Germany), methanol 96% with $\rho = 0.792$ kg/L, potassium chloride p.a. (Phillips Harris, UK), sodium hydroxide p.a. (Farco Chemical) and hydrochloric acid ($\rho = 1.19$ kg/L).

Main devices used consisted of Fourier transform infrared (FTIR) (Shimadzu, Japan), mini scanning electron microscopy Tabletop TM3030 (Hitachi, Japan), conductometer (Mettler

Toledo series LE703, UK), pH meter (Mettler Toledo, UK), bitec-300 incubator (Shimadzu, Japan).

Reagent preparation: Poly(vinyl alcohol (PVA) 10% was prepared by dissolving 10 g of PVA in demineralized water until the volume became 100 mL while heated at 90 °C for 3 h until the PVA was dissolved. Crosslinker solution was prepared by mixing glutaraldehyde 1.25%, sulfuric acid 10%, acetic acid 10% and methanol 50% with the volume ratio of 1:1:2:3 while stirred until homogeneous. A KCl solution 1.0 M was prepared by dissolving 37.28 g of KCl powder in demineralized water until the volume became 500 mL. Meanwhile, 0.5 M KCl solution was prepared by dissolving 18.64 g of KCl in demineralized water until the volume became 500 mL.

Synthesis of PVA/GA hydrogel: As much as 60 mL of PVA 10% was mixed with 60 mL of crosslinker solution (GA) in a beaker glass, stirred using a magnetic stirrer while heated at 55 °C for 5 min. The formed solution was then pipetted and casted on the mold. To fill the mold, as much as 120 mL of mixture was required.

Residual leaching from hydrogel: A total of 100 hydrogel beads were soaked into 100 mL demineralized water in a beaker at 25 °C which had been paired with a pH meter and a conductometer while being stirred using a magnetic stirrer at 200 rpm. The pH and conductivity values of media were recorded every 30 s until the pH and/or conductivity of the solution had not changed for the last 6 min. Stirring was stopped, then the hydrogels were removed and drained, then washed again with demineralized water. The washing was repeated until the pH and conductivity values of the solution (solvent) were constant or relatively constant. After all, the hydrogel was taken out, dried and stored in an incubator, weighed every day until the mass was constant.

Characterization: The characterization of the hydrogels consisted of the instrumentation of functional group and surface morphology of four prepared materials, namely PVA, PVA/GA, PVA/GA-KCl (1.0 M), PVA/GA-KCl (0.5 M) using Fourier transform infrared (FT-IR) (Shimadzu, Japan) and mini scanning electron microscopy Tabletop TM3030 (Hitachi, Japan).

Determination of release behaviour: The work in this section was divided into 2 stages, the first was the KCl impregnation into the gel and the second was the desorption of KCl from the gel. The impregnation of KCl into PVA/GA was carried out by immersing the PVA/GA hydrogel into 1.0 M KCl and 0.5 M KCl solutions. A total of 40 hydrogels were immersed in 500 mL of 1.0 M KCl solution at ± 25 °C, stabilized in a water bath, stirred at 200 rpm for 48 h, removed and dried. The same treatment was carried out by the 0.5 M KCl solution for the other sample of PVA/GA. The impregnated hydrogels were called PVA/GA-KCl (1.0 M) and PVA/GA-KCl (0.5 M).

The release behaviour profile was determined by measuring the quantity of KCl released from PVA/GA-KCl (1.5 M) and PVA/GA-KCl (0.5 M) to aqueous medium with pH 6, pH 7 and pH 8 as a function of time, where the pH of media was adjusted by adding a solution of HCl or NaOH. As much as 9 pieces of PVA/GA-KCl (1.0 M) beads were weighed, then put into the equipped beaker. The values of pH and conductivity of the medium were recorded at 0, 5, 10, 25, 40, 60 and 90 min, both

for PVA/GA-KCl (1.0 M) and PVA/GA-KCl (0.5 M) as the sources of released KCl.

RESULTS AND DISCUSSION

Leaching of residual from hydrogel: Residual leaching from PVA/GA hydrogel was one of main concern in this work for several reasons, *i.e.* (1) there is always a possibility that equilibrium condition was reached during leaching, causing the residual remain exist inside the gel; and (2) to avoid any possible instrumental defects due to artifact caused by the remaining residual. Considering that the synthesis of this hydrogel involved an acidic solvent in the preparation of the crosslinker solution, the possible existence of residual acid was the major concern. This was the reason why the changes in the pH of the medium during washing had become an indicator for the change in the quantity of the residual. In addition to measurement of the pH parameter, it had been also measured the conductivity of the medium in the same system. The pH and conductivity values of medium *versus* time profile of the PVA/GA leaching is shown in Fig. 1.

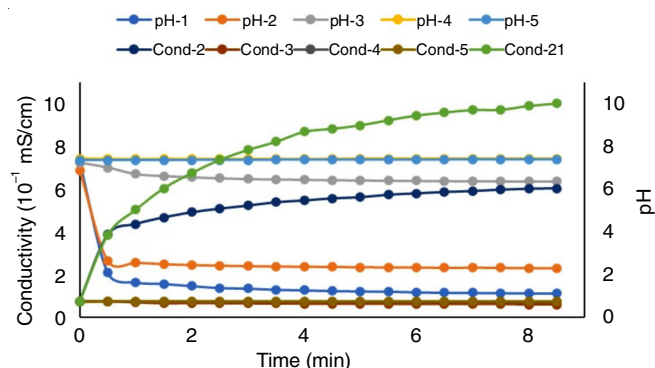


Fig. 1. pH and conductivity of medium as function of time at residual leaching of PVA/GA

Fig. 1 showed that in the first leaching there was a significant decrease in pH and a significant increase in the conductivity values of the leaching medium. A similar phenomenon occurred in the second leaching, but with only smaller change in pH and conductivity values, compared to the first leaching. In the third leaching, the decrease in pH was very small and so as in the increase in conductivity. At the fourth and fifth leaching, the change the pH and conductivity values were negligible since the first minute of observation.

The drastic decrease in pH at the first leaching indicates that the quantity of the acid component in the washed hydrogel was enormous. Similarly, the subsequent leaching process revealed that the hydrogel still contained a significant amount of acid content. However, with just two times of leaching the acid content can be leached almost completely. This was indicated by the negligible decrease in the pH of the washing medium during the third leaching. This explanation was confirmed by the fourth and fifth leaching, where the pH values at this last two leaching stages were practically identical. Practically, washing the hydrogel 5 times with the leaching time of 8 min for each washing was able to remove the dirt or the residual from inside the hydrogel.

In term of conductivity, an increase in conductivity value occurred sharply in the first leaching at first 0.5 min, then slowed down with time, but still showed a significant increase until the end of the first leaching time, which was 8 min. A sharp increase in conductivity also occurred in the second leaching, which lasting during the first 0.5 min of washing, but then sloped and showed a very low increase in conductivity. Meanwhile, in the 4th and 5th leaching there was almost no change both in pH and conductivity value of the leaching medium during the washing time, indicating that the impurities or ionic residues were not leached any longer or the hydrogel was practically free of ionic impurities.

It can be observed that there was a good agreement between the pH and conductivity values in the 4th and 5th washing. These results were in contrast with the phenomenon in the first and 2nd washing, which still showed an increase in conductivity even though the pH did not decrease any longer. Both of these phenomena indicated that there must be non-protonic ions (which affect pH changes chemically) dissolved into the washing medium during the remaining time. Referring to the materials used in the hydrogel preparation, the possible ion sources contributing to the conductivity and pH values are derived mainly from acetic acid and sulfuric acid. The increase in the conductivity value when the pH value is stable can be related to the competition of the supply of hydrated ions from inside the hydrogel into the medium. In this case, the desorption rate of the hydrated hydrogen ions must be faster than that of the hydrated anions that are not bonded to their protons. The effective hydrated radius of hydrogen ions (2.82 Å) is smaller than that of hydrated sulfate ions (3.79 Å) [22], so that the desorption rate of hydrated sulfate ions is slower than that of hydrated protons. Consequently, to achieve charge balance in the washing media, the desorption of sulfate ions into the media continues until the charge balance is reached, which consequently should increase the conductivity value of the medium without changing the pH value.

FT-IR studies: To understand the structural consequences of crosslinked PVA by GA and the addition of KCl to the PVA/GA matrix, the samples of hydrogels were characterized using FT-IR instrument. In Fig. 2, the upper two spectra showed the general profile of PVA and PVA/GA hydrogel, which exhibit the absorption peaks at 3382, 2941.2, 1656.7, 1438.8 and 1095.0 cm^{-1} indicating the presence of functional groups O-H, C-H stretching, C=O, C-H bending and C-O, respectively. Although there were more similarities between the PVA and PVA/GA spectra, however, there was a slight distortion at the wavenumber of 1656.7 cm^{-1} . There was a weakening of the peak intensity around the C=O stretching area and at 1095 cm^{-1} the strengthening of the -C-O absorption intensity.

The addition of KCl into the PVA/GA matrix generally did not change the absorption pattern except in the wavenumber region between 3500 to 3000 cm^{-1} , namely at 3160 cm^{-1} . Another interesting phenomenon was that the absorption intensity for 0.5 M KCl impregnation was greater than that for 1.0 M KCl impregnation. The appearance of a new peak in the 3160 cm^{-1} wave number region was possible due to the interaction between -OH and -Cl, weakening -O-H bonds and the formation of H

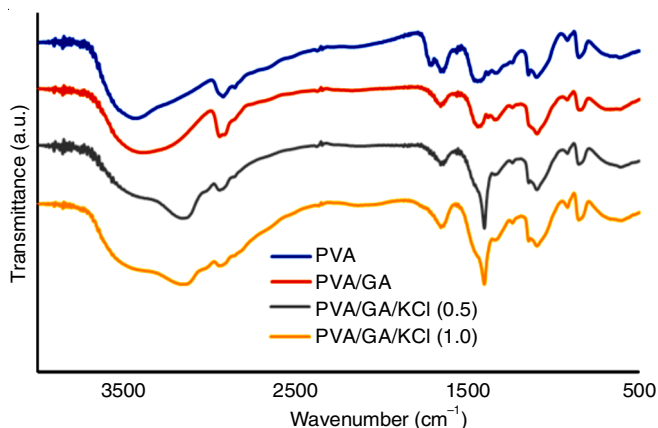


Fig. 2. FT-IR spectra of PVA, PVA/GA, PVA/GA-KCl (1.0 M), PVA/GA-KCl (0.5 M)

and Cl interactions with the formation of $-O\cdots H\cdots Cl$ interactions, known as molecular complexes [23-27]. The FT-IR results indicated that glutaraldehyde (GA) effectively cross-linked PVA and chloride from KCl affected the existence of -OH groups in PVA/GA.

SEM studies: The SEM images of PVA/GA, PVA/GA-KCl (1.0 M), PVA/GA-KCl (0.5 M), at magnification 1000 time both for surface and cross-sectional capture are presented in Fig. 3. The surface image for PVA/GA was looked very smooth, while that for PVA/GA-KCl (1.0 M) and PVA/GA-KCl (0.5 M) were rough. The surface roughness of PVA/GA-KCl (1.0 M), PVA/GA-KCl (0.5 M) seem to be the result of KCl coverage. There was no difference in surface coverage between 1.0 M KCl and 0.5 M KCl, hence 0.5 M was sufficient in this circumstance. In the cross-sectional result, it appeared that the inside of PVA/GA was very clean. Meanwhile, the inner surface of PVA/GA-KCl (1.0 M) and PVA/GA-KCl (0.5 M) seem to be filled with other material, which was suspected to be KCl, where the quantity of KCl inside the PVA/GA-KCl (1.0 M) (Fig. 3.3b) was higher than in PVA/GA-KCl (0.5 M) (Fig. 3.2b).

Release behaviour of KCl from PVA/GA-KCl (1.0 M): Release behaviour test was carried out for PVA/GA-KCl (1.0 M) into the media with initial pH 6, 7 and 8, with the release profile as presented in Fig. 4. The quantity of KCl desorbed was measured conductometrically. This method was considered possible because the PVA/GA that used as KCl host had been washed.

Fig. 4 showed that in the PVA/GA-KCl (1.0 M), the released KCl (in term of conductivity) was very regular within the pH of the applied medium. The released KCl at pH 8 occupied the highest rate, followed by at pH 7 and 6. In the first 5 min of observation, the desorption was rapid, which was indicated by the steepness of curve, with the highest steepness occurring when desorption was carried out on the medium under conditions of pH 8, followed by pH 7 and then pH 6. After 5 min of desorption, the steepness of the curve then decreases and continues to slope until plateau state is reached (at 60 min). This phenomenon implies that the total release is consisted of three process stages, namely the initial phase, the transition phase and the pure desorption phase. In the initial phase, wetting process happened instantly, which was indicated by the occur-

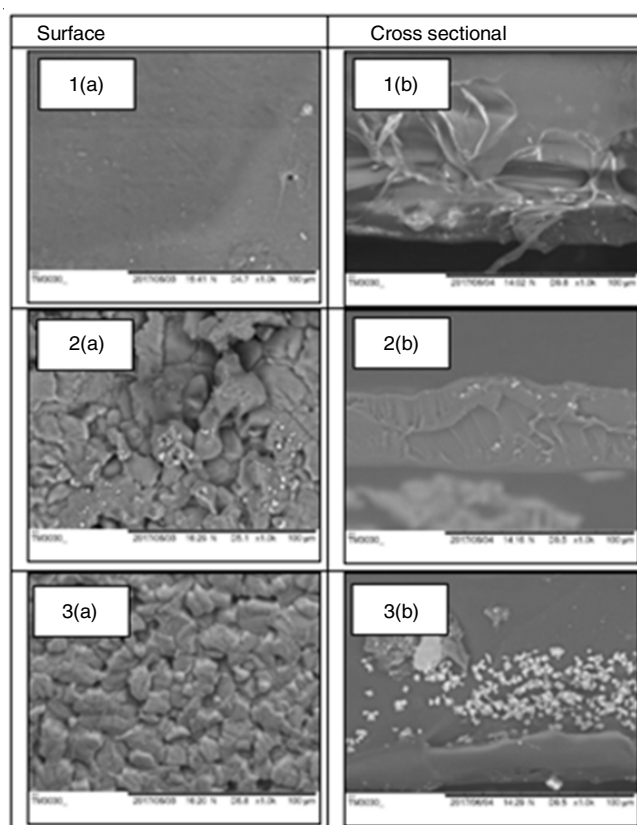


Fig. 3. SEM image of PVA/GA, PVA/GA-KCl (1.0 M), PVA/GA-KCl (0.5 M) at enlargement 1000 time

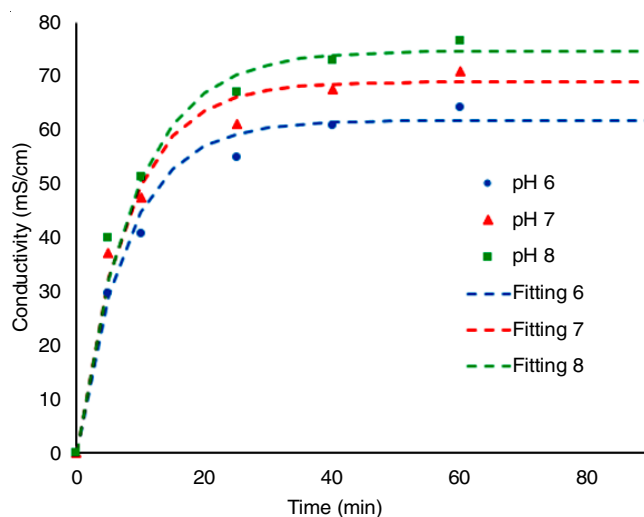


Fig. 4. Conductivity of media as function of time for PVA/GA-KCl (1.0 M) at various pH

rence of dissolution of KCl into the media was happen since the beginning of immersion. This phenomenon indicates that at this early stage, the dissolution of attached KCl onto surface of the gel occurred. While the KCl on the surfaces are being dissolved, water intruded into the gel, hydrated the KCl and desorbed as hydrated ions into the bulk phase. At the beginning of second stage, there was a decrease in the steepness of the curve because of the decrease of KCl concentration on the surface of the hydrogel. The rate of KCl release in the second stage is contributed by two processes, namely cleaning of KCl

on the surface and desorption of KCl from inside the hydrogel. Over time, the KCl desorption contribution has surpasses the dominance of the surface KCl leaching contribution. At the first 30 min, it seems that the contribution of surface KCl leaching has been completed so that the release is fully contributed by the desorption of KCl from inside the hydrogel.

The desorption mechanism has been hypothesized to have first-order kinetics. The initial concentration of potassium chloride (KCl) in the hydrogel is denoted as C_0 at time $t = 0$. If all the KCl completely released into medium then the medium will have the value of the media concentration at infinite desorption time, M_∞ . After desorption takes place ($t = 0$), the concentration of KCl in the hydrogel is $C_t = C_0 e^{-kt}$, where k is the desorption rate constant. The concentration of KCl in the media is M_t , so the value of $M_\infty = C_t + M_t$. By rearranging these expressions, an following expression was obtained for calculating the concentration of KCl in media, as given in eqn. 1:

$$M_t = M_\infty (1 - e^{-kt}) \quad (1)$$

Fitting the conductivity value to time, using the Python 3.8.3 programming language, resulted that the KCl release profile from PVA/GA(1.0 M) at various pH mediums follows first order kinetics (eqn. 1), with parameter values are given in Table-1.

| TABLE-1 FITTING PARAMETER OF THE CURVES IN THE Fig. 5 USING THE eqn. 1 | | | |
|--|-----------------------|------------------------|--------|
| pH | M_∞ (mS/cm) | k ((mS/cm)/s) | R^2 |
| 8 | 74.73505 ± 2.008 | 0.12793 ± 0.01458 | 0.9861 |
| 7 | 68.91722 ± 1.971 | 0.127898 ± 0.01551 | 0.9842 |
| 6 | 61.76316 ± 1.7316 | 0.112711 ± 0.01148 | 0.9871 |

Table-1 shows that the obtained values of M_∞ and k are good fitted to the data, indicated by the value of R^2 in each plot. The interesting feature is that the value of k increases with the increases in M_∞ with a large value of R^2 .

The possibility of correlating the increase in the value of k with the increase in the value of pH was disproved by an argument based on the effect of primary salts on the kinetics of ions in solution. Considering that the desorption rate is of the first-order kinetics and increases with increasing pH, the difference in the desorption rate of KCl in different medium at different acidity conditions does not appear to be due to the primary salt effect. The value of the interaction rate constant of a neutral solvent with a charged solute is not affected by the ionic strength of the environment, as can be inferred from eqn. 2:

$$\log k = \log k_0 - 2AZ_A Z_B \sqrt{I} \quad (2)$$

which shows if one or both Z_A and Z_B are zero then the value of k will be the same in various ionic strengths [28].

In this context, the rate of desorption must be controlled by solvent-solute interactions, namely ion-hydration processes, both on the surface and inside the gel, which follow the mass transfer with chemical reaction (MTWCR) model. Since there is an abundance of water available to hydrate ions, KCl ion concentrations are probably the determining factor in the reaction rate. In this framework, the kinetics of KCl desorption

from PVA/GA into the medium should be a pseudo-first order kinetics. The initial concentration of KCl determines the rate of desorption and the value of the quantity of desorbed material. To support this explanation, a KCl desorption experiments had been carried out from PVA/GA-KCl (1.0 M) and PVA/GA-KCl (0.5 M) into medium with the same acidity conditions, namely pH 7.

Fig. 5 showed that the KCl desorption rate from PVA/GA KCl (1.0 M) was greater than that from PVA/GA-KCl (0.5 M). Likewise, the quantity of desorbed KCl, which was represented by the conductivity value, at the same time, shown that the concentration of KCl originating from PVA/GA-KCl (1.0 M) was always higher than that originating from PVA/GA-KCl (0.5 M) within the time of observation. The plot of conductivity *versus* time for the PVA/GA-KCl (0.5 M) desorption system give the value of $M_\infty = 36.10037 (\pm 1.632)$ mS cm^{-1} and the value of $k = 0.1012936 (\pm 0.01606)$ mS $\text{cm}^{-1} \text{s}^{-1}$ with $R^2 = 0.9701$.

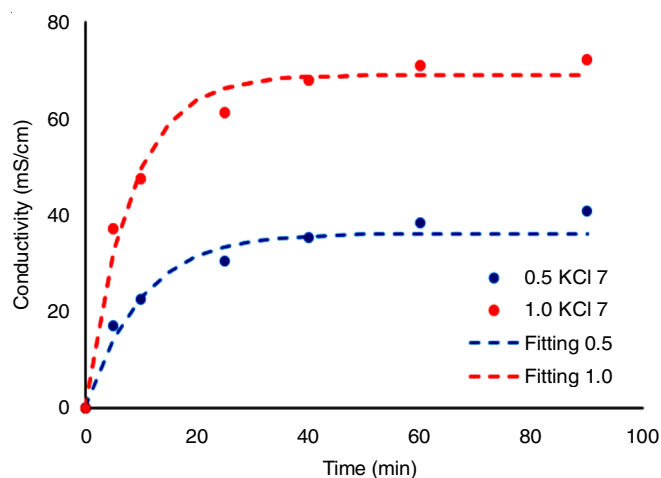


Fig. 5. Conductivity of media as function of time for PVA/GA-KCl (0.5 M) and PVA/GA-KCl (1.0 M) at pH 7

Conclusion

In order to get residual free poly(vinyl alcohol)/glutaraldehyde (PVA/GA) hydrogel, it is necessary to leach the material until the pH of leaching media is neutral and there is no longer change in the conductivity. This condition can be achieved by carrying out leaching processes at least five repetitions, with leaching time approximately 8 min for each. The impregnation of KCl into the PVA/GA matrix causes an interaction between chloride ions and -H on the -OH alcohol group in the PVA/GA matrix by forming a molecular complex. The KCl solution with a higher concentration is able to deposit more KCl into the gel matrix, with a ratio that is almost the same as the concentration ratio of the impregnation medium. The KCl desorption kinetics into the medium constitutes a pseudo-first order kinetics, which is controlled by the amount of KCl in the PVA/GA matrix, with rate constant values (k) being 0.112711, 0.127898 and 0.12793 mS $\text{cm}^{-1} \text{s}^{-1}$ for PVA/GA-KCl (1.0 M) into media pH 6, 7 and 8, respectively. Meanwhile, the k value for desorption of KCl from PVA/GA-KCl (0.5 M) into media pH 7 is 0.1012936 mS $\text{cm}^{-1} \text{s}^{-1}$. The kinetics of KCl desorption from the PVA/GA into media follows the mass transfer with chemical reaction

(MTWCR) model, with water as the extractant and ions from KCl as solute.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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