

Investigating the Semi-batch Extraction of KHCO_3 from Polyamide-6 Pellets

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Abstract

The extraction of potassium bicarbonate from the microporous polyamide-6 pellets feed using a semi-batch extractor has been investigated. The effects of solvent flow rate, solvent temperature, and feed mass on the extraction of solute have been analyzed. As solvent rate increased, the conductivity of miscella increased, and at highest solvent rate of 160 mL/min a maximum solute concentration of 1.9 wt. % KHCO_3 was achieved. The maximal concentration of 2.3 wt. % KHCO_3 was observed at a solvent temperature of 318 K at a batch processing time of 120 seconds. The conductivity of miscella also increased with increased feed masses. Further, the solute concentration in the miscella increased with increased feed masses, and a maximum solute concentration of 2.1 wt. % was achieved at a feed mass of 0.13 kg during the initial stage of extraction. Use of the best operating conditions will optimize the extraction of potassium bicarbonate from polyamide-6 pellets feed on a large scale

Keyword: Polyamide-6; Potassium bicarbonate; Miscella; Operating conditions.

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1. Introduction

Many chemical processes are characterized by an industrially important solid-liquid extraction. The feed is contacted with a solvent in order to separate the desired component. The solid and the solvent remain in close contact, and the desired solute can diffuse from the solid to the liquid, which leads to a separation of components initially present in the feed [1]. The solute diffuses from the solid feed into the nearby solvent. Either the leached solid or insoluble solid, or both, may be a valuable product [2]. The solid-liquid extraction, also known as leaching, is extensively used in food-processing industries. Solids (feed) may undergo considerable change in their characteristics during the leaching operation [3].

The rate of extraction process is influenced by several factors. First, the size of feed particles is pertinent. More specifically, the range of feed particle size must be small enough that every particle requires approximately the same extraction rate. The solvent chosen should be highly selective and must possess low enough viscosity to ensure that it flows easily. The solubility of the solute to be extracted from solid feed increases with increased temperature thus, yielding a higher extraction rate.

Moreover, the diffusion coefficient increases with increased temperature, hence improving the extraction

rate. Solvent agitation is industrially important because it leads to increased eddy diffusion, thereby increasing the transfer of solute from the surface of feed particles to the bulk of the solution. The success of solid-liquid extraction and the techniques to be employed will vary depending on the pretreatment of feed samples [4]. A comparative study of potassium bicarbonate extraction from polyamide feed using batch and single-stage continuous process has been conducted by Danish et al. 2016 [5]. It was concluded that the batch extractor performed better than a continuous one. Poirot et al. 2007 studied the counter-current continuous and batch extractions using vegetable substrate and concluded that the ratio of solid to liquid decreased by a third compared with usual conditions [6]. Jokic et al. 2010 studied the effect of the solvent type, temperature, and extraction time on the extraction of total polyphenols from soybeans. The maximum extraction yield was achieved at 353 K using 50% aqueous solution after 120 minutes of batch time [7]. Spigno et al. 2007 further studied the effects of different operating conditions on the extraction yield [8]. A study [9] devoted to the extraction of oil from jatropha seeds using microwave treatment and ultrasound assisted methods has been carried out and it was suggested that these methods have positive effect on the yield. The significance of current study is to analyze the influence of operating parameters on the extraction of potassium bicarbonate from porous polyamide pellets and the findings of the study may be used to suggest the best operating condi-

tion to optimize the extraction process.

2. Materials and Methods

2.1. Chemicals

KHCO_3 and polyamide pellets (average size 2.68 mm) from Sigma-Aldrich were employed to prepare the feed for extraction. Distilled water was used as a solvent to carry out extraction of the potassium bicarbonate extraction.

2.2. Experimental Set-Up

Fig.1 is a set-up diagram of the solid-liquid extraction unit procured from Armfield (U.K.) that was utilized for conducting the experimental work. The acrylic extraction unit is configured as a batch extractor, and flow rates of the solvent for extraction were adjusted using peristaltic pumps.

Temperature controller was used for fixing the desired set point temperatures of the solvent. The temperature of the solvent and the solute concentration in terms of wt. % KHCO_3 of extracted potassium bicarbonate in miscella were monitored by a temperature sensor and flow-through conductivity probe.

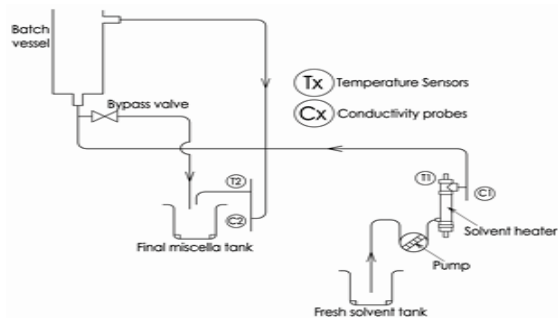


Fig.1. Set-up diagram of solid-liquid extraction unit

2.3 Experimental Procedure

Microporous polyamide-6 pellets containing potassium bicarbonate were used as feed material. First, microporous polymer pellets were dried completely using an oven set no higher than 333 K. Next, 500 grams of potassium bicarbonate was dissolved into 1.5 L of distilled water at 293 K, using a 3-liter batch of pellets (~400 gm) for one run as it was convenient to handle in a 5 L capacity graduated beaker. After this, the solution of KHCO_3 was added to 3 liters of dried polyamide-6 pellets (equivalent to 3 litres of volume, ~400 gm), and the mixture was stirred until the pellets were com-

pletely wetted out with the solution.

In order to absorb the solution, the polyamide-6 pellets were left for almost 24 hours and the mixture was stirred regularly to dispense the solution evenly. The wetted pellets were spread out on the drying trays and dried in an oven set no higher than 333 K. The dried samples were weighed, and their composition (51.5% by weight) was determined and a little amount of prepared KHCO_3 solution remains unabsorbed. Similar batches of feed were prepared for each run to investigate the effect of operating conditions on extraction. The peristaltic pump was calibrated so that exact solvent flow rates could be adjusted and controlled. The conductivity of the (miscella) was monitored and recorded using conductivity meter at regular intervals. The developed pictorial relationship (Fig.2) between the different KHCO_3 concentrations (wt.%) and conductivity (S) was used to tabulate the solute concentration in miscella.

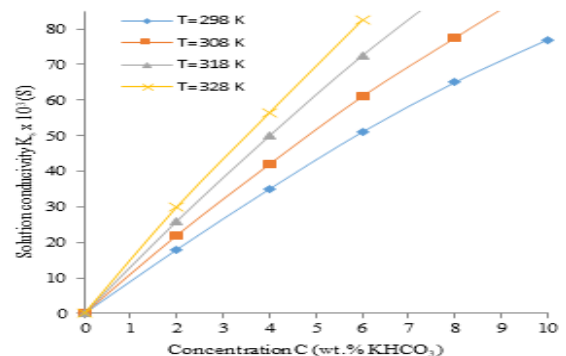


Fig.2 Conductivity vs solute concentration curves

3. Results and Discussion

3.1 Calibration Curve

The peristaltic feed pump was calibrated so that the actual solvent flow could be related to the setting of the rotary speed control. Calibration curve of flow rate Q (mL/min) versus speed setting R was used to adjust and control any desired solvent flow rate.

3.2 Solvent Flow Rates

Superior characteristics of pores play a significant role in the absorption of KHCO_3 into the porous pellets. Higher pore density and pore volume contribute to more absorption of extractable material from the solution in the porous pellets. Firstly, in the process of leaching, the solvent permeates through the bed of feed. Then, the extractable material on the surface is easily washed off by the solvent and thereafter, the KHCO_3 holdup within the porous feed is continuously

dissolved by the solvent. Thereafter solute transfers through pores in the pellet to outside of particle by diffusion. At the end, the extractable material at the surface of porous feed diffuses into the steady layer of the solvent in feed particles and finally mixes with bulk of the solution.

The data were collected at a constant solvent temperature of 298 K with a feed mass of 0.11 kg. The solvent flow rates of 100 mL/minute, 130 mL/minute, 160 mL/minute, and 190 mL/minute were selected to investigate the effect of solvent flow rate on solute extraction. The dependence of miscella (extract) conductivity on batch time at different flow rates is shown in Fig. 3.

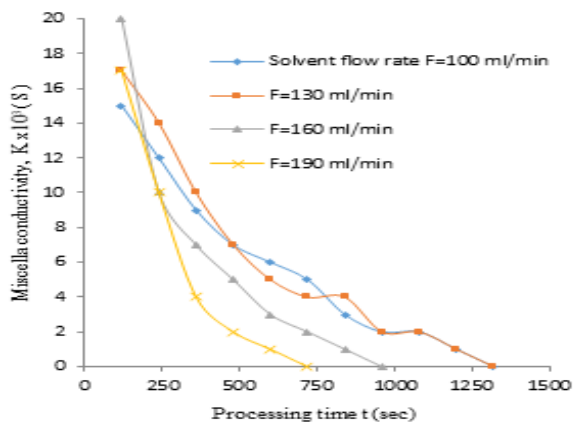


Fig. 3. Dependence of conductivity on time at different solvent flow rates

The maximum miscella conductivity of 20×10^{-3} S was observed at a batch extraction time of 120 seconds with a solvent rate of 160 mL/minute during the initial period of extraction. Conductivity decreases with increased batch processing time until the complete removal of extract from the feed samples. The minimum conductivity of 15×10^{-3} S was recorded during the initial period at a selected solvent flow rate of 100 mL/minute. Batch processing time required to reduce the conductivity to almost zero decreased with increased solvent flow rate within the studied range. The batch processing required to reduce the conductivity to zero S decreases from 1320 seconds to 720 seconds with the increased solvent flow rate from 100 mL/minute to 160 mL/minute. A batch processing time of 960 seconds at a solvent flow rate of 160 mL/minute was required to reduce the miscella conductivity to zero.

The dependence of solute concentration in terms of wt. % KHCO_3 is shown in Fig. 4. Solute concentration in the extract increases with increased solvent flow rate from 100 mL/minute to 130 mL/minute and then decreases at a flow rate of 160 mL/minute. The batch time required to extract all KHCO_3 decreases with increased solvent rate. The highest solute concentration

of 1.9 wt. % KHCO_3 was obtained with a solvent flow rate of 160 mL/minute at a batch processing time of 120 seconds; this decreased to 1.1 wt. % KHCO_3 at a batch process time of 240 seconds.

The extraction rate of solute decreases with increased batch processing time because the amount of solute left in the feed decreases as the process progresses towards completion. The complete removal of extract requires a batch time of 120 seconds at a solvent flow rate of 190 mL/minute compared with a batch processing time of 1320 seconds at a solvent flow rate of 130 mL/minute.

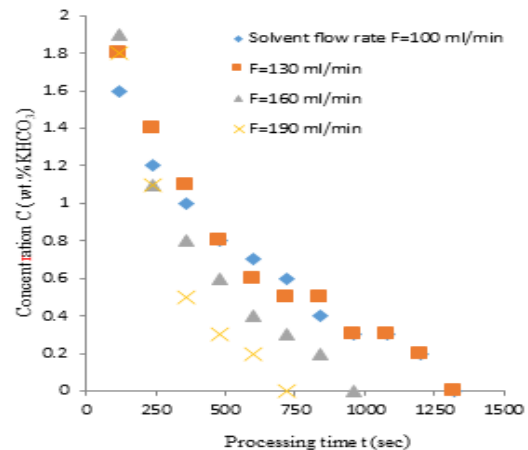


Fig. 4. Dependence of solute concentration on batch time at different solvent flow rates

3.3 Solvent Temperatures

All data were collected at a solvent flow rate of 100 mL/minute and with a feed mass of 0.11 kg. Four different solvent temperatures—298 K, 308 K, 318 K, and 328 K—were chosen to study the effect of temperature on extraction efficiency. The dependence of miscella conductivity on batch processing time is shown in Fig. 5. The conductivity of miscella decreases with increased batch processing time until a solvent temperature of 318 K and then decreases with batch time.

The maximum miscella conductivity of 27×10^{-3} S at a solvent temperature of 318 K and the least conductivity of 21×10^{-3} S under a constant solvent temperature of 298 K were recorded during the initial period of extraction at a batch time of 120 seconds. Higher initial conductivity values are the indicators of a large amount of solute in the fresh feed samples, and conductivity values are reduced to zero after a batch processing time of 1560 seconds for all cases of different solvent temperatures. Investigators concluded that miscella conductivity increases with increased temperature up to a solvent temperature of 318 K and conductivity declines at a solvent temperature of 328 K.

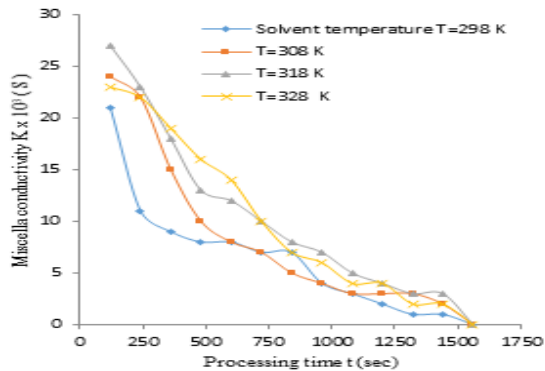


Fig.5. Dependence of miscella conductivity on time at different solvent temperatures

The variation of KHCO_3 concentration with batch processing time is shown in Figure 6. The amount of potassium bicarbonate in miscella declines with increased batch processing time. High solute concentrations of miscella were achieved during the initial period of extraction at a batch time of 120 seconds, because a significant amount of solute was initially present in the feed at all solvent temperatures.

The rate of extraction depends on the amount of extractable material remaining in the feed. The maximum solute concentration of 2.3 wt. % was obtained with a solvent temperature of 308 K at a batch time of 120 seconds, and concentration decreased to 1.5 wt. % at a batch processing time of 480 seconds. Solute was removed completely from the feed samples at a batch processing time of 1560 seconds. At all solvent temperatures, higher extraction rates were observed initially; this is due to the fact that the rate of extraction depends on the amount of solute left in the feed. Higher initial solute concentration in the feed samples leads to a higher extraction rate of solute from the feed.

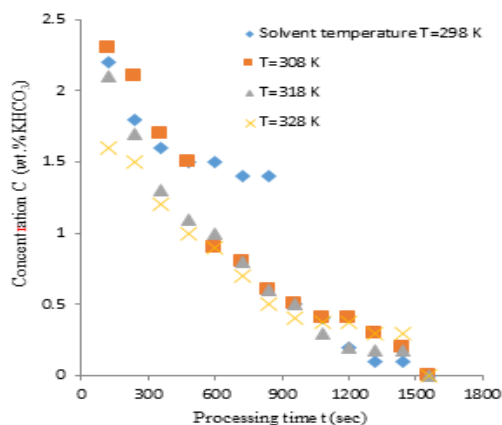


Fig. 6 Dependence of solute concentration on batch time at different solvent temperatures

3.4 Effect of Feed Masses

Data were collected at a solvent temperature of 298 K with a solvent flow rate adjusted to 100 mL/minute. The variation of miscella conductivity with batch processing time is shown in Fig. 7. The conductivity of miscella decreases with increased batch processing time, and almost complete removal of extract was achieved at batch processing periods of 960 to 1080 seconds. Higher conductivities were recorded during the initial period of extraction under all conditions of different feed masses.

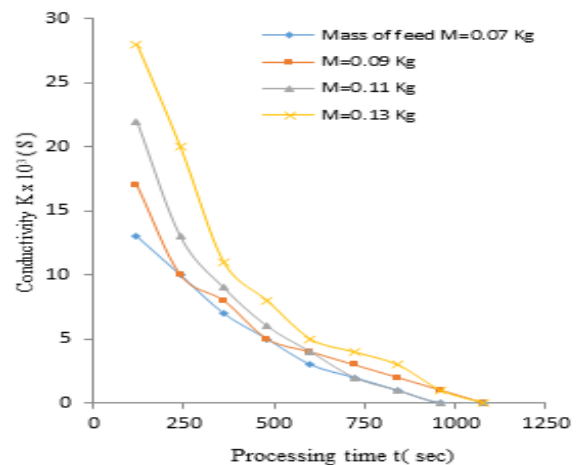


Fig.7. Dependence of miscella conductivity on processing time at various feed masses

Miscella conductivity increases with increased feed masses. Miscella conductivity increases from 13 S to 28 S on increasing the feed mass from 0.07 kg to 0.13 kg at a batch processing time of 120 seconds during initial stage of extraction. Increased mass of feed sample owing to high solute concentration contributes to higher miscella conductivity. Miscella conductivity decreases from 22 S to 13 S with increased batch time from 120 seconds to 240 seconds at a feed mass of 0.11 kg.

Fig.8 shows the dependence of wt. % KHCO_3 concentration on batch processing time under different conditions of feed masses. Higher extraction rates were achieved during the initial period of extraction. The solute concentration in miscella decreases with increased batch time as concentration of the KHCO_3 declines with the progression of the process, whereas the solute concentration in miscella increases with increased feed mass due to increased mass of KHCO_3 in the feed.

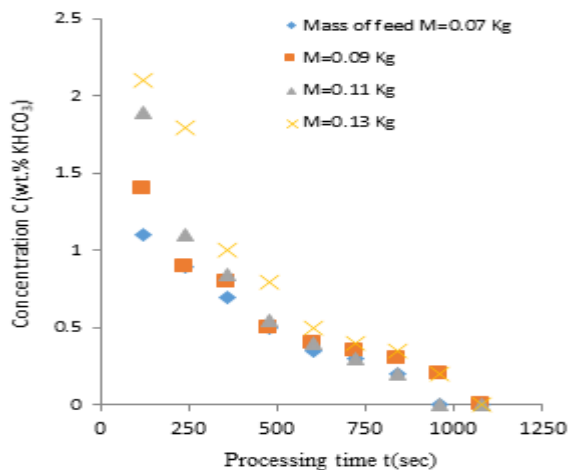


Fig. 8 Dependence of concentration on processing time at various feed masses

The increase of feed mass leads to increased solute material in the feed samples. The highest concentration of ~ 2.1 wt. % KHCO_3 was reached at a feed mass of 0.13 kg compared with a concentration of 1.9 wt. % at a feed mass of 0.11 kg during the initial period of extraction at a batch time of 120 seconds.

Sodium bicarbonate concentration of ~ 1.1 wt. % was obtained with a feed mass of 0.07 kg at batch processing time of 120 seconds, and the complete removal of solute was achieved after a batch time of 960 seconds. It was found that extraction rates decline with processing time due to decreased mass of extractable material in feed. With all feed masses, almost complete removal of extractable material was achieved at the end of batch processing periods of 960 to 1080 seconds.

4. Conclusions

The conductivity of miscella (extract) increases with increased solvent flow rate up to a solvent rate of 160 mL/minute. Its maximum value is 20×10^{-3} S at a solvent flow rate of 160 mL/minute and decreased to 17 S at a solvent flow rate of 190 mL/minute. The batch processing time required to extract the solute decreases with increased solvent flow rate. Therefore, it is not always desirable to select a high solvent flow rate. The maximum sodium bicarbonate concentration of 1.9 wt. % was achieved with a solvent rate of 160 mL/minute at a batch time of 120 seconds compared with other selected solvent rates. The miscella conductivity increases with increased temperature from 298 K to 318 K, and its maximum value was recorded at a temperature of 318 K.

Under the conditions of different solvent temperatures, high extraction rates were achieved during the

initial period of extraction. The highest sodium bicarbonate concentration of 2.3 wt. % in miscella was obtained at a solvent temperature of 308 K. High solute concentrations in the fresh feed samples lead to higher extraction rate during the initial stage of the extraction process. The conductivity of miscella increases with increased feed masses, and maximum conductivity of 28×10^{-3} S was recorded for a feed mass of 0.13 kg at a batch processing time of 120 seconds. High extraction rates were achieved during the initial period of extraction under different conditions of feed masses. The solute concentration of miscella increases with increased feed masses. The findings of the current research work may be used to optimize the extraction of potassium bicarbonate from polyamide-6 pellets from feed.

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