

Extraction of KHCO_3 from Microporous Polyamide 6-Pellets using Batch and Single-Stage Continuous Process

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Abstract

The extraction of KHCO_3 from microporous polyamide 6-pellets feed containing potassium bicarbonate has been investigated experimentally. The batch and single-stage continuous processes are used for comparing the extraction efficiency. High purity distilled water has been used as solvent for the extraction of KHCO_3 . Experiments were conducted at solvent temperature of 45°C and solvent flow rate was adjusted to 110 ml/min. Conductivity of miscella for batch extraction decreased drastically with processing time and finally reduced to zero. Almost complete removal of solute (%wt KHCO_3) from the porous feed containing KHCO_3 was achieved after a processing time of about 22 minutes in case of batch extraction. In case of single-stage continuous extraction conductivity of solute in miscella decreased gradually and then attained almost steady-state value during single-run period. Lesser removal of solute KHCO_3 was noticed during continuous extraction. It was inferred that batch extractor performed better compared with single-stage continuous extractor. Results obtained may be used to suggest the more suitable extraction process either batch or continuous which may be more suitable for a specific application.

Keywords: Extraction, Polyamide, Conductivity, Solvent.

Introduction

Numerous chemical industrial processes are characterized by an important solid-liquid extraction process. Inorganic/organic substances found in a mixture of different components in a solid. The feed (solid) is contacted with a liquid phase (solvent) in order to segregate the desired solute or remove an undesirable solute from feed (solid phase). The two phases (feed and solvent) remain in close contact and the desired soluble component (solute/solutes) can diffuse from the solid to liquid phase that leads to a segregation of the components initially in the solid¹.

This extraction/leaching process requires the transfer of a soluble component (solute) of a solid material utilizing a liquid solvent. The solute diffuses from the solid feed material into the surrounding solvent. One of these, either the leached solid or the insoluble solid (also known as spent solid) or both may be the desired and valuable product of the process². Leaching is widely used in natural products, metallurgical, and food industries. The process is termed as washing when an undesirable components is segregated from feed mass by utilizing water as solvent.

The amount of material recovered in leaching is usually greater than in ordinary filtration washing and it varies extremely from the washing of filtered solids. During leaching operation, solids may undergo substantial change in characteristics³. The static solid-bed extraction is accomplished in a tank vessel with a pierced false bottom which is used to support the solids and allow the drainage of the solvent. Solids (feed) are laden into

tanks, sprinkled with solvent till the soluble component (solute) exists in the solid is decreased to the required level. In some industrial application, extraction rate is so quick that one time circulation of the solvent through the feed material is sufficient, but the countercurrent-flow of solvent through a series of extraction tanks is more usual industrially.

There are four salient factors that influence the extraction rate⁴. Thus if the solute diffusion through the permeable structure of the residual is the controlling factor, the material to be used as a feed should be of small size so that the solute has to travel a small diffusion path length.

Particle Size: The rate of leaching/extraction is greatly affected by size of particles in numerous ways. The small particles contribute to significant interfacial area between the solid and the liquid and this enhanced area therefore leads to higher rate of mass transfer of desired solute. It is generally advantageous that the range of particle sizes must be very small so that every particle needs approximately the same extraction time.

Solvent: The liquid chosen should be highly selective and must possess considerably low viscosity that may provide ease in flow of the solvent. Relatively pure solvent is used initially, but as the extraction proceeds the concentration of the solute increased and extraction rate decreases gradually, firstly due to reduced concentration gradient and secondly owing to increased solution viscosity.

Temperature: Largely, the solubility of the solute being

extracted from feed increases with increased temperature to yield higher extraction rate. Furthermore, the diffusion coefficient is increased with rise in temperature and hence, improves the rate of extraction.

Agitation of the fluid: Solvent agitation is industrially important because it leads to increase the eddy diffusion and hence increases solute transfer from the surface of particles to the bulk of solution. Further, the sedimentation of fine particle suspensions is prevented by agitation.

The success of solid-liquid extraction and the techniques to be utilized will very frequently depending upon any pre-treatment of solid feed. In most of the cases, small particles of the soluble material are completely enclosed in a matrix of insoluble components. The solvent first diffuse into the solid mass and the resulting solution containing the solute then diffuse from, before a separation can result⁵.

Size reduction techniques such as crushing and grinding of feed material will greatly increase the rate of extraction, because the soluble portions of the feed are made more approachable to the solvent. Because of cellular structure of vegetable and animal bodies, the natural products (solute) to be extracted from these materials are found inside the cells. In order to prepare the feed for extraction, the sugar beets are chopped into thin slices termed as cossettes before process so as to lessen the processing time needed for solvent water to approach the each and every plant cells. The vegetable seeds/ beans like soybeans are normally peeled or rolled to give particles in size range 0.15 to 0.5 mm. The cells generally smaller than this size are mostly fractured by the flaking process, and the then oils (solutes) are more easily contacted by the solvent.

Vegetable substrates were utilized to investigate the solid-liquid extraction in counter-current continuous extractor and a batch study was conducted to acquire the bulk-phase saturation and kinetics of extraction⁶. The solid-liquid ratio has been decreased by a third compared to usual conditions. The change of ratio decreases the solvent amount used and its regeneration. Technique for batch to continuous transposition and operating parameters' optimization has been proposed in this investigation.

The impact of the solvent, extraction time and temperature on the batch extraction has been studied⁷. The investigation was carried out in order to select the most solvent for obtaining the highest yield of total polyphenols from soybeans. The results showed a significant effect of the solvent and temperature on extraction yield and the kinetics.

The effect of operating conditions like temperature, pH, sampler solid ratio and the extraction time of leaching the phenol compounds using feed (barley) has been studied⁸. The solid-liquid proportion of 30 ml/g was used to model the extraction of polyphenolic compounds using most successful solvent.

Materials and Methods

Chemicals: Potassium bicarbonate (KHCO_3) obtained from Sigma-Aldrich and polyamide 6 pellets were used to prepare the feed for solid-liquid extraction. De-mineralised water is used as solvent for solid-liquid extraction because hard water (tap water) may form precipitates and hence it may affect the conductivity measurement.

Experimental Set Up: Solid-liquid extraction unit obtained from Armfield (U.K.) has been used for carrying out the experimental work. The extraction unit is configured as a single stage for continuous operation and separate vessels made of acrylic is provided which allows batch extraction to be performed. The batch of feed is placed in a cloth bag to avoid the transportation of polyamide. The flow rates of the solvent either for batch or continuous operation can be adjusted using peristaltic pumps. PID temperature controller is used for adjusting the solvent temperature. The solvent temperature and concentration of the extracted potassium bicarbonate in the solvent are monitored by a temperature sensor and flow-through conductivity probe.

Experimental Procedure: Microporous polyamide 6-pellets were used a porous material. This material is an inert and plastic material in the pellet form that provides a suitable porous carrier for the extractable material. First, the porous polymer pellets are dried completely using oven set to no more than 60°C. Three liters batch of dry pellets were used and 500 gm of extractable material dissolved into 1.5 liters of water at 20°C (as per solubility data). After this solution of KHCO_3 was added to 3 liters of dried polyamide-6 pellets and mixture was stirred until the pellets were completely wetted out with the solution.

Polyamide pellets were left for almost 24 hrs to absorb the solution and mixture was periodically stirred to dispense the solution evenly. The wetted pellets were spread out on the drying trays and dried using the oven set to no more than 60°C. The dried samples were weighed and composition was determined. Due to low density of the pellets, the ratio is nearly 1:1.8 i.e. 500 gm in total weight of 900 gm of porous polymer pellets and corresponding concentration is 55%. The peristaltic pumps were calibrated so that the exact solvent flow rates can be adjusted.

Results and Discussion

Batch extraction process: Approximately 100 gm of prepared feed was used and temperature of the solvent water was adjusted to 45°C to carry out the extraction of KHCO_3 . The fresh solvent (water) at a volumetric flow rate of 130 ml/min is fed into the batch extractor continuously and the extract (miscella) leaving the process is monitored regularly. Conductivity of the extract as it exits the batch vessel is measured at regular interval so as to monitor the extraction of KHCO_3 from the feed material. The dependence of conductivity with time is presented in Figure-1.

It requires a batch time of 22 minutes to extract all the KHCO_3 from the feed. Conductivity of miscella was reduced to zero after a period of 22 minutes as no more solute was present in the feed.

The dependence of concentration of solute (% wt. KHCO_3) with processing time under batch conditions is represented in Figure-

2. It is evident that extraction rate was highest during initial period of extraction at a process time of 4 minutes. After attaining a maximum extraction rate, it starts decreasing and it was noticed that decrease of solute concentration is gradual. Complete removal of solute in terms of %wt. KHCO_3 was achieved after batch processing time of 22 minutes.

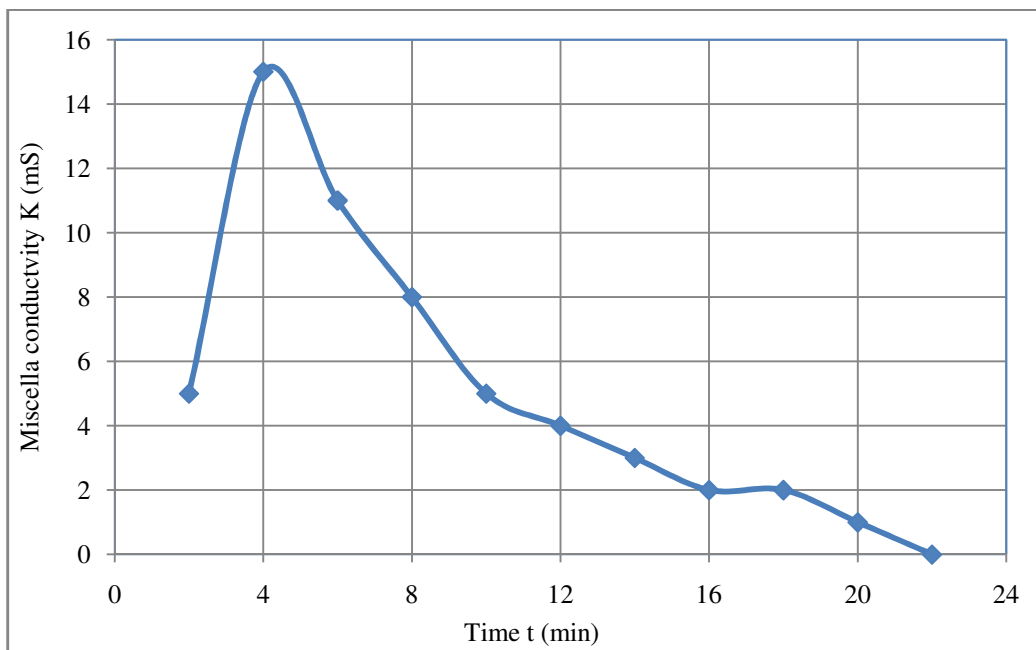


Figure-1
Miscella conductivity versus time curve for batch process

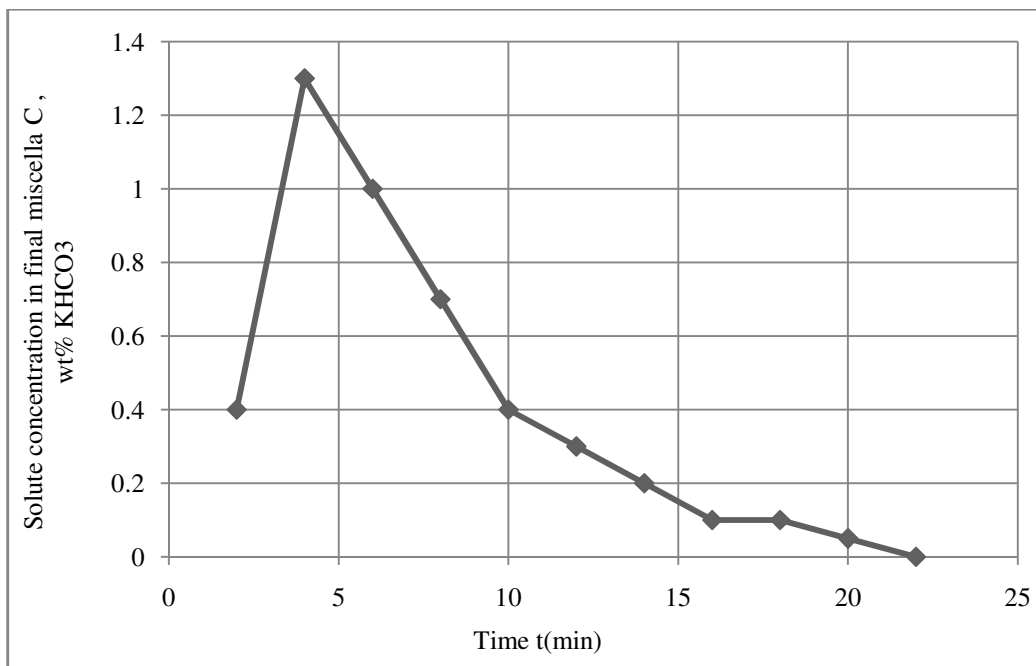


Figure-2
 KHCO_3 concentrations versus time curve for batch process

Single-stage continuous extraction: Sufficient amount of feed was prepared for performing the experiment when configured for continuous extraction as single stage and solvent temperature was adjusted to 45°C. The fresh feed is fed into the system using spiral material feeder. The solid material is moved through the system by the rotating cell and the fresh solvent sprinkled onto it regularly drains through it. Once the solid material has been processed it discharges into the spent solids tank. The dependence of miscella conductivity with time for single stage continuous extraction is presented in Figure-3. Data were collected for a single run lasting up to approximately 20 minutes.

The rotational speed of the rotor was adjusted to 2 rev/min and the speed of the spiral feeder was controlled to give the required depth of material in the cell (40-50%). The single stage extraction is an open loop system and stabilizes after an initial period. The maximum value of conductivity 10 mS was recorded during initial period of extraction and after that it attains a almost constant value throughout remaining run period of an experiment. In case of batch extraction process, conductivity of miscella almost reduced to zero but this is not the case with single-stage extraction highlighting the higher degree of extraction associated with batch extraction process.

The dependence of extracted KHCO_3 concentration with single run time of 20 minutes is presented in figure-4. Percentage weight of extracted KHCO_3 in final miscella was determined using conductivity vs. time curves. More potassium bicarbonate (%wt KHCO_3) upto 0.9 wt% was extracted during initial stages of extraction and after that rate of extract declines and solute concentration remains constant throughout the remaining run period ranging from 0.6-0.7 Wt% KHCO_3 .

Batch vs. continuous extraction: The dependence of extracted potassium bicarbonate (%wt KHCO_3) with time for batch extraction and single-stage extraction is presented in figure-5. During the initial period of extraction, the more KHCO_3 was extracted for batch process compared with single stage extraction. It is evident that at a processing time of 4 min, concentration of solute in the miscella was 1.3 % wt KHCO_3 for batch process as compared with a lower solute concentration of 0.9% wt. KHCO_3 .

Almost 100% of solute removal of KHCO_3 was achieved after a period of around 22 minutes processing time for batch extraction but in case of single- stage continuous extraction, steady-state extract concentration of 6-7%wt KHCO_3 was maintained during the single run period.

Conclusion

After initial adjustment, conductivity of miscella decreased drastically with batch processing time and finally reduced to zero. Complete removal of solute (KHCO_3) from the porous feed containing potassium bicarbonate was achieved after a processing time of about 22 minutes in case of batch extraction. In case of single-stage continuous extraction conductivity of solute in miscella decreased gradually and then attained almost steady-state during remaining run period. Lesser removal of solute KHCO_3 was noticed during continuous extraction. It was concluded that batch extractor performed better with compared to single-stage continuous extractor. Results obtained may be used to suggest the solid-liquid extraction process more suitable for an application.

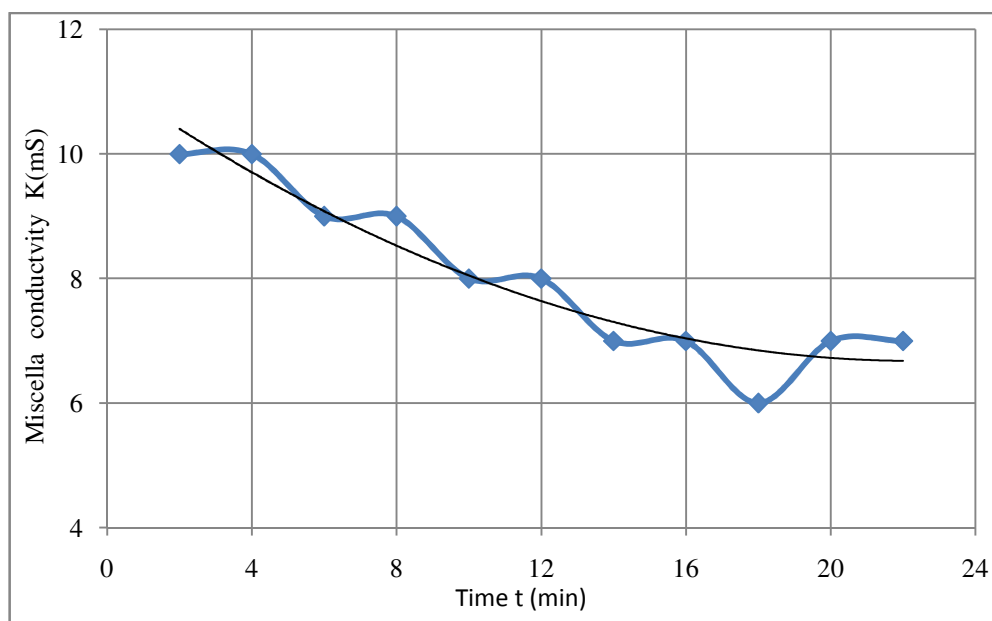


Figure-3
Conductivity versus time curve for single-stage continuous extraction

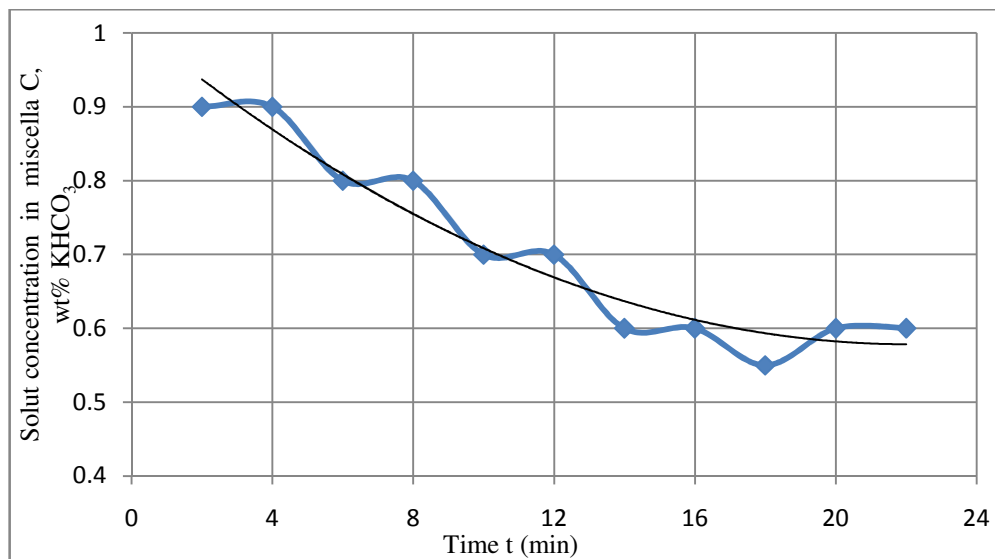


Figure-4
KHCO₃ concentrations versus time curve for single stage continuous extraction

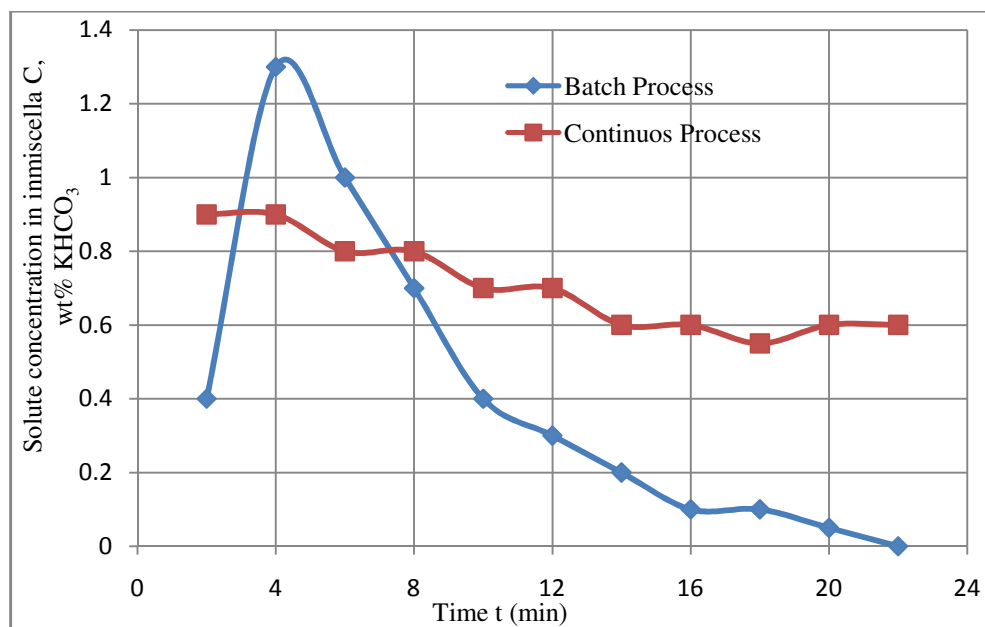


Figure-5
% Wt. KHCO₃ versus time curve for batch and continuous type extraction

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