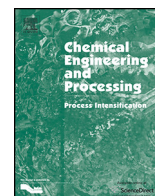




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Development of a unique modular distillation column using 3D printing



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ABSTRACT

3D printing is recently used in many fields of technology, from medical science to aerospace and food products. In some cases, this technique is faster and increases the design flexibility in comparison with conventional machining. In this article, the design and manufacturing of a modular distillation column are presented using 3D printing. The flexibility and freedom in 3D model designs and the challenges for developing a small scale distillation column are demonstrated. The column was designed in a coil shape with modular structure to overcome the printing area limitation of the used 3D printer. The chemical compatibility of the printed polymer with several common solvents was examined. A 3D printable packing, and a commercially available stainless steel spring packing were used in the distillation. The column was used to distill a binary mixture of hexane + cyclohexane in full reflux and continuous mode. The heat loss of the column was also measured at the boiling points of hexane and cyclohexane. The composition of reflux and boilup streams were analyzed online using a gas chromatograph equipped with two sampling valves. The temperature of the vapor phase was measured at the top (vapor outlet) and bottom (boilup) of the column.

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1. Introduction

3-Dimensional (3D) printing technology (additive manufacturing) has developed rapidly during recent years. This method makes it possible to manufacture complex components economically. This method provides more flexibility in the design and manufacturing of mechanical parts compared to the traditional machining technologies. Now, this technique is used in medical applications such as hearing aids [1,2], prototyping of electrical [3], or mechanical parts such as vehicle body production [4], building constructions [5], aerospace industry [6] and even food printing [7]. However, the application of 3D printing in chemical research and development is more challenging. This can be due to the harsh environment, such as high temperature - pressure or incompatibility of the polymeric 3D printing materials with some chemicals. Some 3D printing methods with higher costs such as Direct Metal Laser Sintering (DMLS) or Laser Powder Forming (LPF) can be used for these kind of conditions [8].

Stereolithography is a 3D fabrication process where a photopolymerizable resin is selectively polymerized to a desired geometric shape in a layer-by-layer fashion [9]. Stereolithography has already found several applications in different engineering fields: e.g. Leigh et al. [10] demonstrated the fabrication of a miniature flow sensor, Lucklum et al. (2015) [11] used polymer stereolithography to manufacture a functioning gas chromatography column and Kanai and Tsuchiya [12] fabricated complex nozzles for monodisperse water/oil emulsion applications.

Distillation is one of the most common methods to purify compounds in chemical industry. Górak and Sorensen [13] mentioned that up to 50% of the capital and operational costs of industrial plants are dedicated to the distillation units. The widespread application of distillation units makes it one of the most mature technologies in the chemical industry, however the research and development on different aspects of distillation is carried out to increase its efficiency or to use it in new processes.

During recent years, many of the research works in distillation technology has been focused on decreasing the size of the pilot scale distillation columns to milli/micro-scale [14,15]. The smaller size of pilot distillation columns makes the manufacturing and modifications of the column faster and easier. Additionally, it reduces the costs for the production and operation of the columns.

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Nomenclature

3D	3 dimensional
CAD	Computer-aided drafting
DMLS	Direct metal laser sintering
GC	Gas chromatograph
LPF	Laser powder forming
MAD	Mean average deviation
N. A.	Not available
N. C.	Not calculated
P & ID	Piping and instrumentation diagram
SLA	Stereolithography
VLE	Vapor-liquid equilibrium
Symbols	
B	Bottom product flowrate, $g\ h^{-1}$
C_p	Isobaric heat capacity, $J(g\ K)^{-1}$
dT_1	Difference between the T_{boilup} and $T_{lit(xV)}$, K
dT_2	Difference between the $T_{vap.out}$ and $T_{lit(xL)}$, K
D	Distillate flowrate, $g\ h^{-1}$
F	Feed flowrate, $g\ h^{-1}$
F_s	Flooding factor, $Pa^{1/2}$
h	Enthalpy, $J\ g^{-1}$
HDT	Heat deflection temperature, K
HETP	Height equivalent of a theoretical plate, cm
ID	Internal diameter, ID
L	Reflux flowrate, $g\ h^{-1}$
L_{column}	Packed height, cm
MAD	Mean average deviation
n	Number of GC runs
NTS	Number of theoretical stages
OD	Outer diameter, mm
Q	Heat duty, W
R_D	Reflux ratio
t	time, min
T	Temperature, K
T_B	Boiling point, K
T_{boilup}	Temperature of the boilup stream measured by thermometers, K
$T_{lit(xL)}$	Regressed temperature from literature at (x_L) , K
$T_{lit(xV)}$	Regressed temperature from literature at (x_V) , K
$T_{vap.out}$	Temperature of the vapor outlet, K
u_s	Velocity of vapor, $m\ s^{-1}$
V	Boilup flowrate, $g\ h^{-1}$
x_L	Average mole fraction of hexane in the reflux stream
x_V	Average mole fraction of hexane in the boilup stream
x_{avg}	Average of all mole fractions measured by GC
x_{GC}	Mole fraction measured by GC
$\Delta h_{vaporization}$	Enthalpy of vaporization, $J\ g^{-1}$
ΔQ	Heat loss, W
ΔQ_{avg}	Average heat loss, W
ρ_v	Vapor density, $kg\ m^{-3}$
Subscripts	
avg	Average
boilup	Boilup
in	Inlet
liq. out	Liquid outlet
out	Outlet
reflux	Reflux
vap. out	Vapor outlet

Also, it increases the safety of the operation. In these studies, different distillation columns were manufactured and their characterization were studied, for example: Foerster et al. [16] and Liu et al. [17] manufactured different types of columns and investigated the methods to measure the compositions of the distillate and bottom product; or Jang and Kim [18] studied the energy efficiency of a horizontal column and compared it with normal vertical columns. Sundberg et al. [19], Stanisch et al. [20] and Kenig et al. [21] reviewed the recent progress in micro-distillation technologies.

The key idea of this work was to study the possibilities of developing new chemical devices by using a 3D printing method. In order to achieve this, we developed a small scale modular distillation column, which can be printed by a Stereolithography (SLA) 3D printer in several pieces. The packing of the column can be printed with the same technique as the column itself or normal laboratory scale packings can be used. In this work, a flange type connection is designed which can be used to add or remove sections based on the demand. This modularity makes it possible to do the modifications rapidly for one section without affecting the rest of the column.

The column characterization including the temperatures in inlets and outlets, heat loss, the flowrates and online compositions of the boil-up and reflux streams were measured during this work. Moreover, in continuous mode, the product compositions, flowrates and mass balances were studied for the continuous mode. The temperatures for the boilup stream and vapor outlet were measured and compared with vapor-liquid equilibrium data from literature.

2. Methods and materials*2.1. Distillation column design and manufacturing*

The main target of this work was the development of a distillation column with the 3D printing method. The Autodesk Inventor® 2016 was used for designing the 3D parts. The SLA 3D printing technology was used for manufacturing the distillation column (See Section 2.3). This method was favorable due to an affordable printing price, rapid manufacturing, wide selection range of printing materials, which can improve the compatibility with the chemical environment, and robust manufactured parts that are reasonably leak proof. However, for the most of currently available SLA 3D printers the build area is limited (In this work, the maximum build area of the 3D printer was $8.5\ cm \times 6.4\ cm$).

The affordable and rapid production process of SLA 3D printing can help the designers to develop several prototypes and choose the most suitable designs to be used in the experimental studies. The procedure flowchart for this work, and the approximate times needed is presented in Fig. 1.

In this work, two designs were developed. The first design which is presented in Fig. 2 was a column with the external size of $5\ cm$ (length) \times $4.5\ cm$ (width) \times $5.6\ cm$ (height). In this design, all of the inlets, outlets and the body of the column was integrated in one piece. This distillation column was developed based on the horizontal distillation column that was previously studied in our research group by Sundberg et al. [22] and in other groups such as Jang and Kim [18]. The column featured a coil shaped channel with height of $0.4\ cm$, width of $1.2\ cm$ and total length of $10.8\ cm$. The bottom of the channel was having cone shaped structures to act as packing. This distillation column had favorable characteristics such as the small holdup ($5.0\ cm^3$), compact structure which could reduce the heat loss, and short printing time (less than 6 h). Despite these benefits, this design was not satisfactory during the laboratory runs. It was noticed that the narrow vapor-liquid channel could be blocked with some layers of unexpected polymer

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