

Improving bench scale design for polymerization of green CO₂-polymers

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Abstract

Plastic has become the most common material since the beginning of the 20th century and has become almost indispensable due to its durability, light weight and low costs. However, some of the characteristics also make the plastic problematic when it comes to the end of its life phase. Norner Research AS, a polymer research institute performing extensive independent or funded research in the plastic value chain from process technology to final product and application, has during the last ten years been active in developing biodegradable green plastic that can replace up to 40% of the fossil raw materials with industrial captured CO₂. The green plastic, poly(propylene carbonate) (PPC), is produced by reacting propylene oxide (PO) with CO₂. The aim of this paper is to study the effect of static mixer on production of PPC and improve the catalytic process by reducing the reaction time and increasing the yield. The process includes multiphase gas-liquid flow, and good mixing is one of the key factors to succeed in improving the process. The flow behavior of the components in the multi-phase bench scale reactor is studied in order to investigate the potential of reducing the reaction time by using static mixers to increase mass transfer. A static mixer is a device for the continuous mixing of fluids, and can be used to mix liquids, gases or to mix a gas into a liquid. The energy needed for mixing comes from a loss in pressure as the fluids flow through the static mixer.

Computational fluid dynamic (CFD) is an important tool in order to simulate and optimize the polymerization process. In this study, the CFD software Ansys/Fluent is used to investigate the multiphase flow through the static mixer. The static mixer is simulated to study the effect on the gas-liquid mixing. The simulations were performed with mixtures of 17% and 34% PPC in PO under CO₂ pressure. The simulations showed that the required number of mixing elements (L) to obtain a uniform mixing of CO₂, PO and PPC in the liquid phase is estimated to be 6.5L for the 17% PPC case and 5.25L for the 34% PPC case. Consequently, using more than the required number of mixing elements would increase the pressure drop without increasing the mixing. It was observed that the required mixer length decreased when the composition of PPC in the inlet liquid phase was increased from 17% to 34%.

Keywords: *Carbon dioxide based polymers, poly(propylene carbonate) green plastic, CFD, Ansys/Fluent, bench scale pilot reactor, static mixer*

1 Introduction

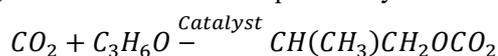
In recent years, the interest in green, sustainable materials to replace more common, unsustainable materials is becoming more and more relevant (Luinstra, 2008). One of the many reasons for this may be the lack of biodegradability of commonly used polymers such as polyethylene (PE) and polypropylene (PP). The stability of these polymers has a negative impact in nature, especially for marine life (Tokiwa et al., 2009). Another central topic nowadays is the CO₂ produced by modern activities and its contribution to the “greenhouse effect”. CO₂ is one of the largest resources of carbon readily available, and several journals suggest that reducing the CO₂ levels in the atmosphere can be beneficial towards halting climate change from human activities (Mikkelsen et al., 2010).

The process of synthesizing PPC from CO₂ was studied and published as far back as 1969 (Inoue et al., 1969). As time passed, varying levels of research was put into this topic. At the start of the 21st. century, climate change by human activities and its relation to increased CO₂ levels in the atmosphere as well as concerns on plastic waste management has sparked a new interest in using CO₂ based polymers (Luinstra, 2008). However, the main focus in these studies were on development of effective catalyst solutions and not much work is reported on the process development (Narang et al., 2016; Kember et al., 2011).

The use of static mixer in a tubular reactor was shown to be advantageous for similar process (Gürtler et al., 2013). In this study the multi phase flow through the static mixer is investigated.

2 Production of green polymer

PPC is produced by the co-polymerization between the greenhouse gas carbon dioxide (CO₂) and propylene oxide (PO) in the presence of a catalyst. Depending on the choice of the catalyst system, temperatures in the range of 50 to 100°C and pressures up to 60 bar are reported in literature (Luinstra, 2008; Coates et al., 2004). The reaction can be expressed by:



The process efficiency (reaction rate and yield) is one of the critical aspects that has to be investigated to obtain a successful production of poly(propylene carbonate). A simplified sketch of the bench scale polymerization process, which is featured in this paper, is illustrated in

Figure 1. The process consists of an autoclave reactor with an associated recycle loop. Valves and measurement sensors are not included in the drawing. However, viscosity, density, temperature, volume flow and pressure measurement sensors are installed in the bench scale rig. The existing bench scale rig does not include a static mixer, and the aim of this paper is to study the effect of installing a mixer. The plan is to mount the static mixer on the vertical pipe in the recycle loop, as shown in the figure, to improve the mixing between CO₂ and PO. The performance of a static mixer is studied in this paper by using computational fluid dynamics (CFD) modelling and simulations.

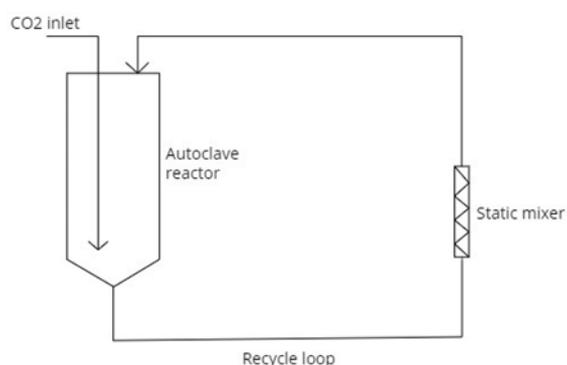


Figure 1. Draft of the bench scale rig for polymerization of poly(propylene carbonate).

3 Static mixer

The process of mixing different phases (gas/liquid, liquid/liquid, liquid/solids) with a static mixer is one of the more widely used mixing processes in chemical industries. Even so, the understanding of flow and mixing patterns within the mixers has been lacking until recently and has mainly been the property of static mixer producers. Several studies proclaim increased reaction yield and higher process efficiency when using in-line static mixers to disperse two or more fluid phases in the laminar regime (Fradette et al., 2006; Won and Kyu, 2004). Fortunately, the former ways of using mainly heuristics and know-how from experience to assess a static mixers performance is being replaced by more scientifically precise and quantifiable methods, as for example CFD (Zalc et al., 2002).

Static mixer will give an additional pressure drop in the polymerization process. The pressure drop in the static mixer can be validated by defining a dimensionless ratio Z based on the pressure drop over the elements

of an in-line mixer divided by the pressure drop of an open pipe of the same length as the mixer. This Z -value represents the increased energy required to push a fluid through a pipe with static mixer elements compared to one without such elements.

$$Z = \frac{\Delta P_{SM}}{\Delta P_{OP}} \quad (1)$$

ΔP_{SM} represents the pressure drop the fluid undergoes from the inlet of the first mixing element to the outlet of the last element in the pipe. ΔP_{OP} is the pressure loss over an open pipe with an equal length, and is calculated from the Hagen-Poiseuille equation:

$$\Delta P_{OP} = \frac{8\mu LQ}{\pi R^4} \quad (2)$$

where μ is the dynamic viscosity, L is the length of the pipe, Q is the volumetric flow rate and R is the pipe radius (Zalc et al., 2002).

In chemical processes where the temperature is a critical factor, installation of a static mixer gives a more uniform temperature distribution in addition to the improved mixing of the chemical components over the cross section area of the pipe. Non-uniform temperature distribution can potentially reduce the yield or degrade the product quality in a chemical reaction process.

There are many different static mixer designs available on the market, and the type of mixer has to be selected based on the fluids involved in the process. For mixing in a polymerization process, the most important is the capability of mixing non-Newtonian pseudo-plastic flow in the laminar regime. Figure 2 shows an image of a static mixer (Fusion Fluid Equipment).



Figure 2. Example of a static mixer (Fusion Fluid Equipment).

4 CFD set-up

This chapter will include the choices made with respect to mesh, model, method and schemes used in the simulations. The programs used for CFD in this paper are available from ANSYS and include ANSYS Design Modeler, ANSYS Meshing, ANSYS Fluent and ANSYS CFD Post. All these programs are combined into what is known as ANSYS Workbench.

4.1 Design and meshing of static mixer

ANSYS Design Modeler was used to import the STEP files of the static mixer. The files are CAD-drawings containing 3D data of the construction of the static mixer. After importing the STEP files to Design Modeler, the program was used to define a gas inlet, a liquid inlet and an outlet. This was done by defining a face at the inlet pipe and a face at the outlet pipe, effectively closing off the internal volume of the geometry. The face at the inlet pipe was split into two halves, defined as the gas inlet and liquid inlet respectively. The inlets were automatically defined as velocity inlets while the outlet was automatically defined as a pressure outlet. This is a default function in Design Modeler, which is applied by using the string “inlet” or “outlet” in the naming of the inlets and outlet respectively. After the inlets and outlet were defined, the fill function in Design Modeler was used to define the internal volume of the mixer. This internal volume represents where the fluids can be located inside the static mixer.

ANSYS Meshing was used to construct the mesh from the internal fluid domain extracted from the geometry in Design Modeler. Mostly, the default settings in ANSYS Meshing were used. CFD and Fluent were chosen as physics preference and solver preference respectively, while the element type was set to tetrahedrons. Tetrahedral elements were chosen over hexahedral and polyhedral because tetrahedrons are better suited for complex geometries. (Phoenix Analysis and Design Technologies, 2017). The size function was initially set to “adaptive” as default, but this was changed since the “adaptive” size function only generated 89000 elements, which resulted in a coarse mesh. The “proximity and curvature” size function generated 10.9 million elements, which was assumed too fine and computationally demanding. Increasing the minimum element size from the default value of 4.2906e-5 meters to 4.2906e-4 meters reduced the number of elements to 3252795 and number of nodes to 644440, which was assumed precise enough, since 3.5 million elements were used in (Zalc et al., 2002). For a mesh to be valid in representing a flow field, it must be of a certain quality. There are several properties, which indicate the quality of the mesh, depending on the element types. Some are general for all element types, such as node distribution and element smoothness. Node distribution is related to boundary layers and are important for areas of the flow where gradients are large, for example near walls. The lack of boundary layers is visible in Figure 3. This is likely one of the major contributors for potential erroneous calculations (numerical diffusion) in the mesh. Element smoothness is related to how large differences there are between the volumes of the elements for adjacent elements.

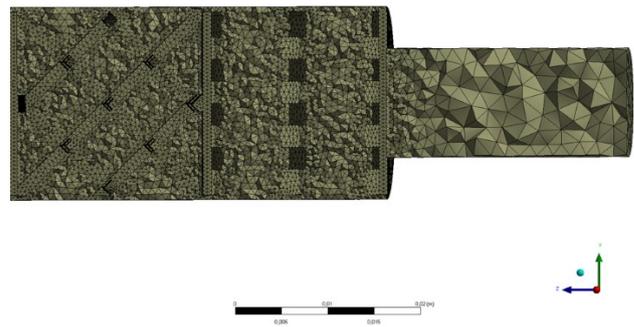


Figure 3. "Dissected" mesh

Ideally, this volume gradient between adjacent elements should be small enough to minimize potential truncation errors from the governing equations. For a tetrahedral mesh such as the one in this paper, element skewness and element aspect ratio are two important parameters. Skewness can be calculated in two different ways, but for tetrahedral elements, the equilateral-volume based skewness is calculated. The difference between the volume of an element and the volume of an equilateral element with the same circumradius is defined as the element skewness, and is expressed as: (ANSYS Inc., 2017)

$$Skewness = \frac{\text{optimal element size} - \text{element size}}{\text{optimal element size}} \quad (3)$$

Skewness is measured between 0 and 1 where 0 is equilateral and 1 is degenerate. There are several intervals between these numbers to categorize and decide if the elements comprising the mesh are good enough. These intervals are listed in Table 1.

Table 1. Skewness values and element qualities (ANSYS Inc.)

Value of skewness	Element quality	
1	Degenerate	
0.9 - < 1	Bad	
0.75 - 0.9	Poor	
0.5 - 0.75	Fair	
0.25 - 0.5	Good	
>0 - 0.25	Excellent	
0	Equilateral	

Degenerate elements will give faulty results as the equations being solved work under the assumption that the elements are approximately equilateral. Diverging calculations are typical because of such elements. A general rule says that a tetrahedral mesh should have a maximum skewness of less than 0.95 and an average skewness of less than 0.33 (ANSYS Inc., 2009). The maximum, minimum and average skewness of the mesh used in this study are presented in Table 2.

Table 2. Mesh skewness data

Minimum skewness	9.98 e-9 ≈ 0
Maximum skewness	0.84
Average skewness	0.24

The aspect ratio is the measure of how stretched an element is. The aspect ratio is defined as the ratio between the distance from an element centroid to a face centroid, and the distance from an element centroid to a node. The highest of these values is divided by the lowest to give the aspect ratio of an element. An aspect ratio of 1 means an equilateral tetrahedron and the higher the number gets, the more stretched out the tetrahedron will be. The aspect ratio of a tetrahedron, Q , is calculated as shown in Equation 4 (Cascade, 2017). The minimum, maximum and average aspect ratio of this mesh is listed in Table 3.

$$Q = \frac{h_{max}}{2*\sqrt{6}*r} \quad (4)$$

where h_{max} is the largest edge length and r is the radius of the tetrahedron.

Table 3. Mesh aspect ratio data.

Minimum aspect ratio	1.1575
Maximum aspect ratio	14.241
Average aspect ratio	1.8755

4.2 Models and input to ANSYS Fluent

After the mesh was validated with respect to skewness and aspect ratio, the next task was solving the discretized algebraic system of equations for the velocity and pressure fields throughout the mesh. This was done using ANSYS Fluent. Several choices had to be made with respect to solver, models, defining materials, boundary conditions, pressure-velocity coupling scheme and spatial discretization schemes. ANSYS Fluent has two different solvers, the pressure-based solver and the density-based solver. Generally, the pressure-based solver has been used for low-velocity incompressible flows while the density-based solver has been used for high velocity, compressible flows. The pressure-based solver was chosen for the simulations in this study. Steady state calculations were chosen because of time limitations and lack of polymerization reactions in Fluent. (Bakker, 2006) Two steady state cases were considered, which was the mixing of CO₂ and PO with 17 % and 34 % PPC respectively. The reason for using these compositions are that former reactor tests have been carried out with the same compositions, and the viscosity data from these tests were available. To different compositions were used to investigate the degree of mixing between CO₂ and PO as the amount of PPC increases in the mixture. Gravitation was included, and the gravitational acceleration was defined as -9.81 m/s² in the z-direction while the fluid flows in positive z-direction. This was

done to simulate that the static mixer is mounted vertically as is intended in the pilot reactor design. Models used in Fluent was the mixture multiphase model and the energy equation. Since both the gas inlet and the liquid inlet is defined with the same velocity, the Stokes number is assumed very low. Another reason is that the particles are gas bubbles, which tend to follow the fluid streamlines well. This should make the mixture multiphase model applicable for use in the simulations. Except for CO₂, which was predefined in the material database in Fluent, both PO and PPC had to be defined prior to the calculations. PO was defined with the properties presented in Table 4.

Table 4. PO properties (National Center for Biotechnology Information, 2018)

Density	830 kg/m ³
Specific heat	2153.93 J/kg*K
Thermal conductivity	0.134 W/m*K
Viscosity	0.00028 kg/m*s
Molecular weight	58.08 g/mole
Standard state enthalpy	-122.6 kJ/mole

Since molten PPC exhibits pseudoplastic/shear thinning characteristics, PPC was modelled with the non-Newtonian power law in Fluent:

$$\eta = k\gamma^{n-1}H(T) \quad (5)$$

n is a measure of deviation from Newtonian fluids. $n < 1$ is shear-thinning, $n = 1$ is Newtonian and $n > 1$ is shear-thickening. k is an average viscosity of the fluid, γ is the shear rate and $H(T)$ is Arrhenius law. Minimum and maximum viscosity for use in the non-Newtonian power law have to be included in Fluent. The power law inputs and other PPC properties are presented in Table 5.

Table 5. The power law inputs and other PPC properties

n	0.6 [19]
k	54.46 [7]
Density	1310 kg/m ³ [20]
Specific heat	1641.2 J/kg*K
Thermal conductivity	Same as for PO (no values were found)
Molecular weight	102 g/mole
Standard state enthalpy	Same as for PO (no values were found)

5 Results and discussion

The simulations were carried out using a mixture of CO₂, PO and PPC. The viscosity of PPC is significantly higher than the viscosity of CO₂ and PO and it is therefore important to include the PPC when simulating the static mixer. Two cases were simulated, one with 17% and one with 34% PPC in the liquid phase. The feed to the mixer included 17% and 34% PPC. The entire static mixer is comprised of 12 x-grid mixing elements. Each element has a length L , which gives the

entire mixing region a length of 12L. Figures 4, 5, 6 and 7 represent the results from the simulation with 17% PPC and show the fractions of CO₂ (to the left), PO (in the middle) and PPC (to the right) at position 1/4L, 1L, 2L and 5.25L respectively. The contour plot color scheme is explained in Table 6.

Table 6. Contour plot color scheme explanation

Color	Approximate volume fractions
Red	1
Yellow	0.75
Green	0.50
Light blue	0.25
Dark blue	0

The inlet to the static mixer is split into two equal parts defined as the gas inlet and liquid inlet respectively. CO₂ is fed into the mixer from the right, and can be observed moving from the right side of the cross-section towards the left. The PO and PPC can be seen emerging from the left side of the pipe cross section. The CO₂ is gradually mixed with the liquids along the length of the mixer.

Figure 6 shows an increase in mixing for CO₂ and PO. Both are still moving towards left and right respectively, but the largest rate of mixing for CO₂ and PO occurs from the top of the pipe towards the bottom and vice versa. This shows that the x-grid mixer mixes the fluids in both axial and radial directions. This gives an effective mixing, but also high pressure loss. The red PPC zones have all been reduced to green and blue, suggesting increased mixing compared to the mixing after 1L (Figure 5). The required mixing length to obtain a uniform mixing of CO₂, PO and PPC is estimated to be 5.25L as shown in Figure 7. Some changes in the mixing patterns from 6.5L towards 12L are observed, but they are minimal. The pressure drop through the mixer is proportional with the number of mixing elements, and to avoid high pressure drop, the number of mixing elements should not exceed the number of elements needed for the required degree of mixing. Using more than 7 mixing elements in this case would increase the pressure drop without increasing the mixing significantly. The pressure drop through the mixer in this case was 2676 Pa, whereas it could be reduced to half of 1561 Pa if a mixer with 7 elements were used.

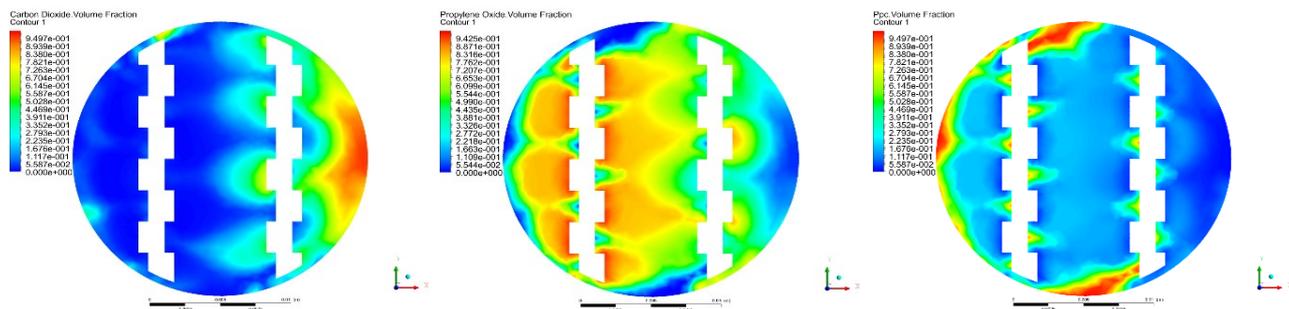


Figure 4. Contour plots of CO₂, PO and PPC respectively at 1/4 L. The feed mixture includes 17% PPC.

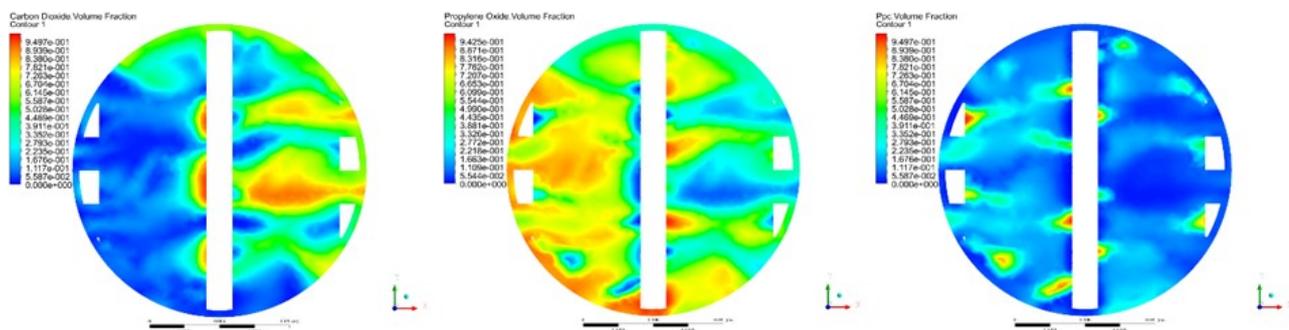


Figure 5. Contour plots of CO₂, PO and PPC respectively at 1 L. The feed mixture includes 17% PPC.

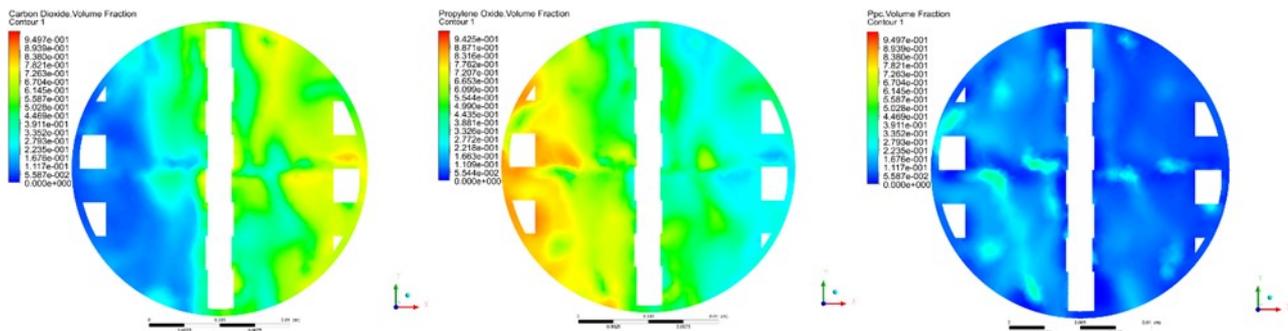


Figure 6. Contour plots of CO₂, PO and PPC respectively at 2L. The feed mixture includes 17% PPC.

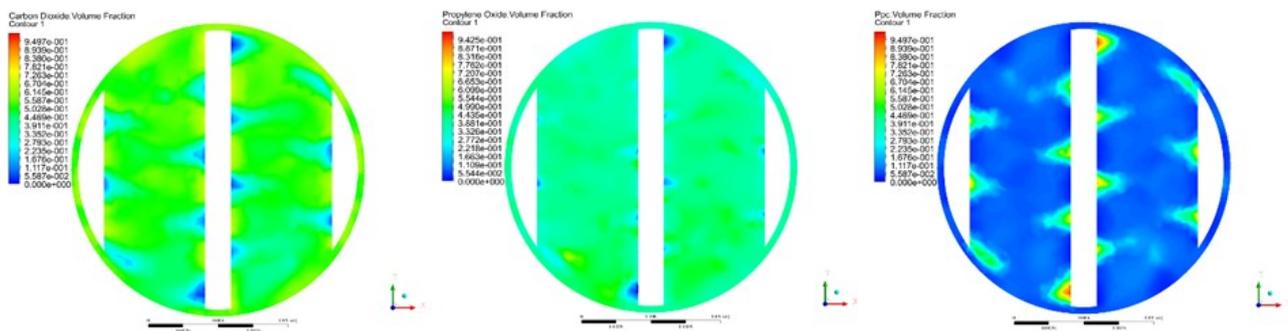


Figure 7. Contour plots of CO₂, PO and PPC respectively at 6.5L. The feed mixture includes 17% PPC

Figure 8, 9, 10 and 11 show the results from the simulation with 34% PPC in the liquid phase. The figures represent the volume fractions of the components CO₂, PO and PPC at the different locations (0.25L, L, 2L and 5.25L) in the static mixer.

As with the 17% PPC case (Figure 4), the 34% PPC case shown in Figure 8 starts off in the same way. CO₂ plumes emerging from the right towards the left. Figure 11 shows the mixer length required to get uniform mixing for the 34% PPC case. As in the case with 17% PPC there are some small differences in the mixing patterns from 5.25L to 12L, but they are too small to justify the increased pressure drop.

As mentioned earlier, the pipe cross section that leads into the static mixer was cut into two halves, the CO₂ inlet and the liquid inlet. This most likely prolongs necessary mixing length simulated in CFD compared to the real process, as both the gas and liquid phase will arrive at the static mixer premixed to some degree. This

ensures that the mixing length estimation for uniform mixing will be conservative. From all the figures, it can be observed that the pseudo-plastic PPC tends to “stick” to the pipe walls and mixing element edges. To conclude, the rate of PPC mixing into the CO₂ + PO mixture compared with the rate of CO₂ mixing with PO is low. The reasons for this can be multiple, but the major factors are probably the lack of polymerization reactions in the Fluent simulations, and the large variations in the viscosity of PPC compared to CO₂ and PO. However, the results show that good mixing will be obtained by reducing the mixing elements to five or six. The pressure drop through the mixer was calculated to 4084 Pa, and by reducing the number of mixing elements to six, the pressure drop will be reduced to 2042 Pa.

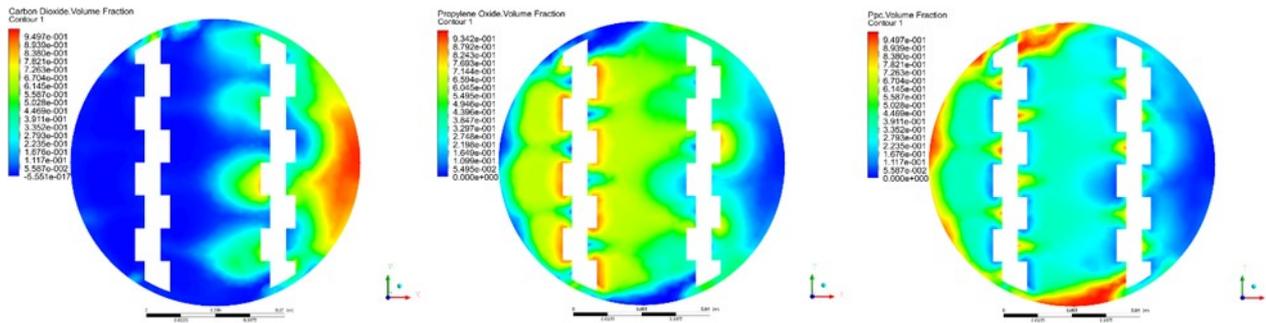


Figure 8. Contour plots of CO₂, PO and PPC respectively at 1/4 L. The mixture includes 34% PPC.

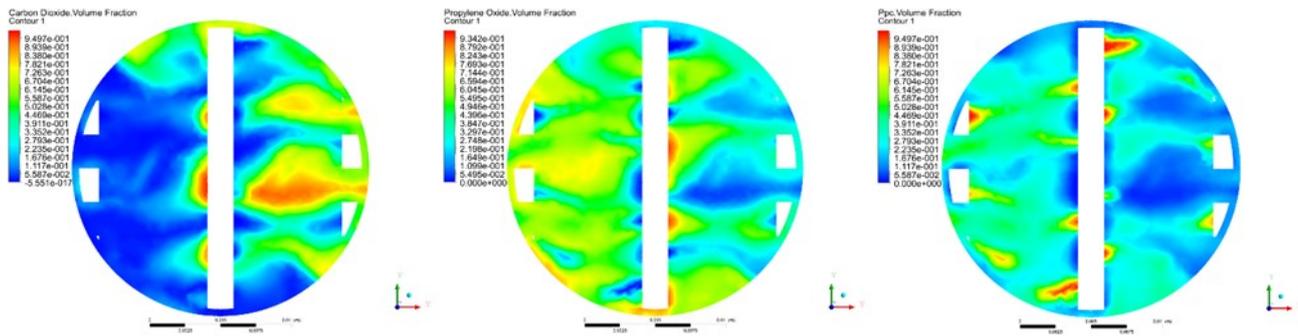


Figure 9. Contour plots of CO₂, PO and PPC respectively at L. The mixture includes 34% PPC.

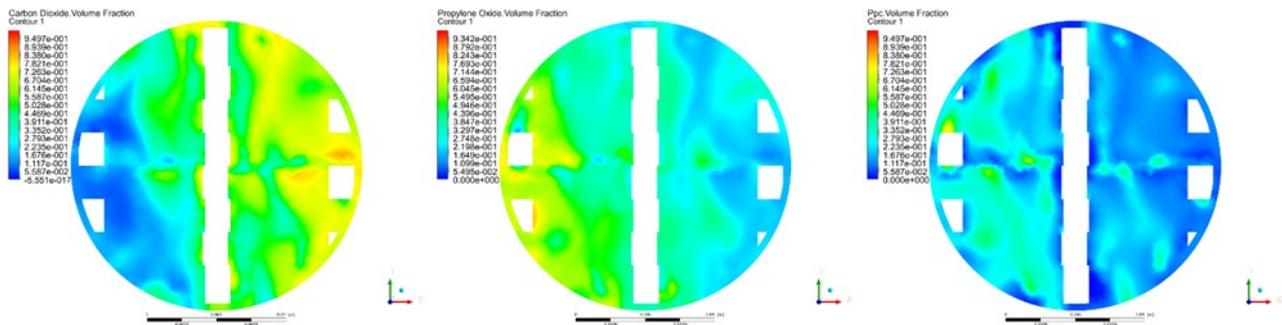


Figure 10. Contour plots of CO₂, PO and PPC respectively at 2 L. The mixture includes 34% PPC.

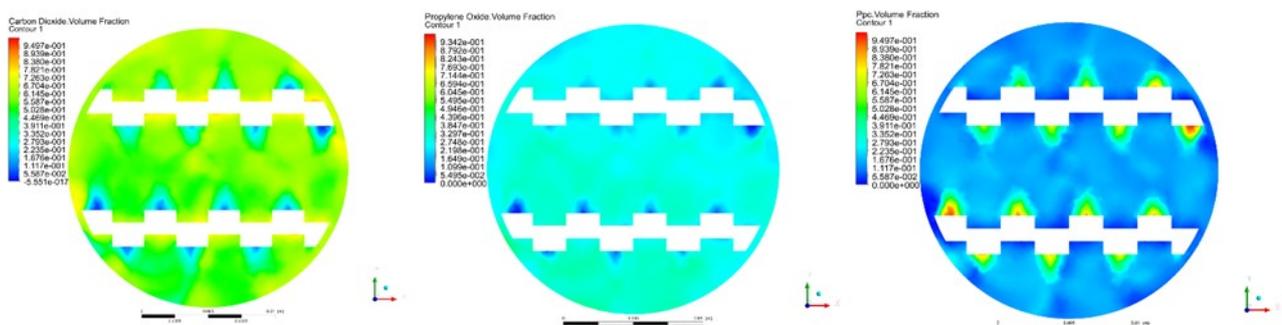


Figure 11. Contour plots of CO₂, PO and PPC respectively at 5.25 L. The mixture includes 34% PPC.

6 Conclusion

Green plastic (PPC) is produced by reacting propylene oxide with CO₂. The process includes multiphase gas-

liquid flow, and good mixing is one of the key factors to succeed in improving the process. The flow behavior of the components in the multi-phase bench scale reactor is studied in order to investigate the potential of reducing the reaction time by using a static mixer to increase mass

transfer. In this study, the CFD software ANSYS/Fluent is used to investigate the multiphase flow through a static mixer with twelve mixing elements. The static mixer is simulated to study the effect on the gas-liquid mixing. The simulations were performed with mixtures of PO and 17% and 34% PPC in the liquid phase and pure CO₂ in the gas phase. The required mixing length to obtain a uniform mixing of CO₂, PO and PPC is estimated to be 6.5L and 5.25L for the case with 17% and 34% PPC respectively. Some changes in the mixing patterns from 6.5L towards 12L are observed, but they are minimal. The pressure drop over the mixer is proportional to the length of the mixer. Using more than seven mixing elements would increase the pressure drop without increasing the mixing significantly. The pressure drop over the mixer was 2676Pa when the liquid feed consisted of 17% PPC and 4086Pa when 34% PPC was used in the liquid feed. A decrease in required mixing elements was observed when the composition of PPC in the inlet liquid phase was increased from 17% to 34%.

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