

MOTIVATION

1. Studies have shown that improving the porosity of the anode catalyst layers of fuel cells, during manufacture, has led to more efficient energy production due to increased mass transfer area.
2. By using lithium carbonate as a pore former in the anode layer manufacture of direct formic acid fuel cells resulted in 25% increase in current density⁴.

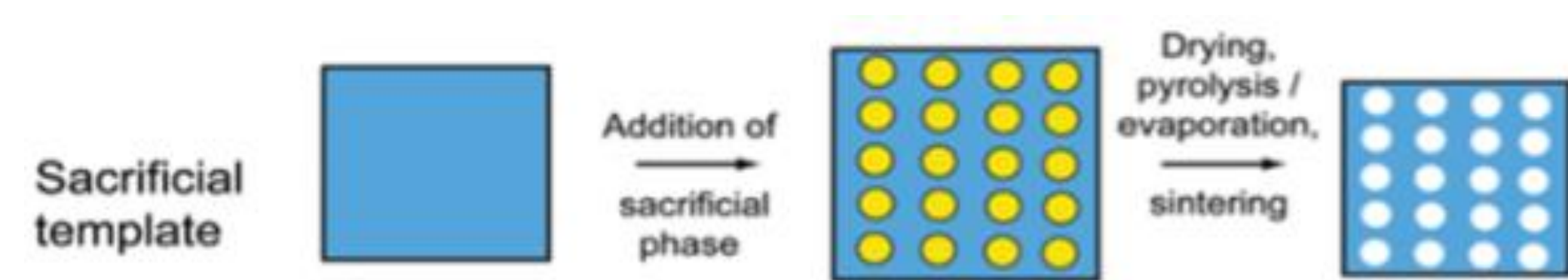


Fig: Schematic of sacrificial template.

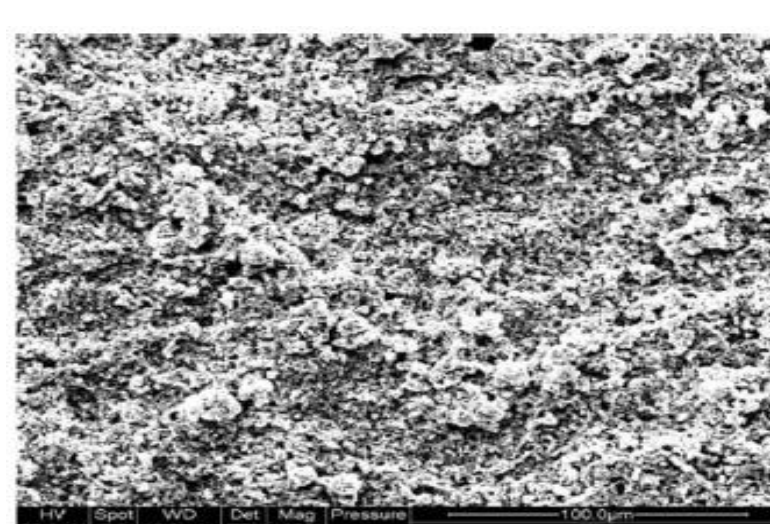


Fig: SEM image of anode layer in the absence of pore-former¹

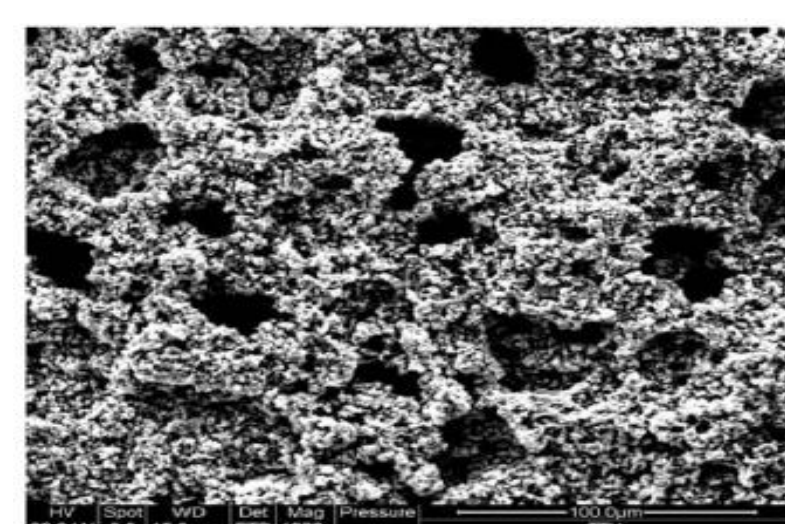


Fig: SEM image of anode layer after use of pore-former¹

APPROACH: Microfluidics

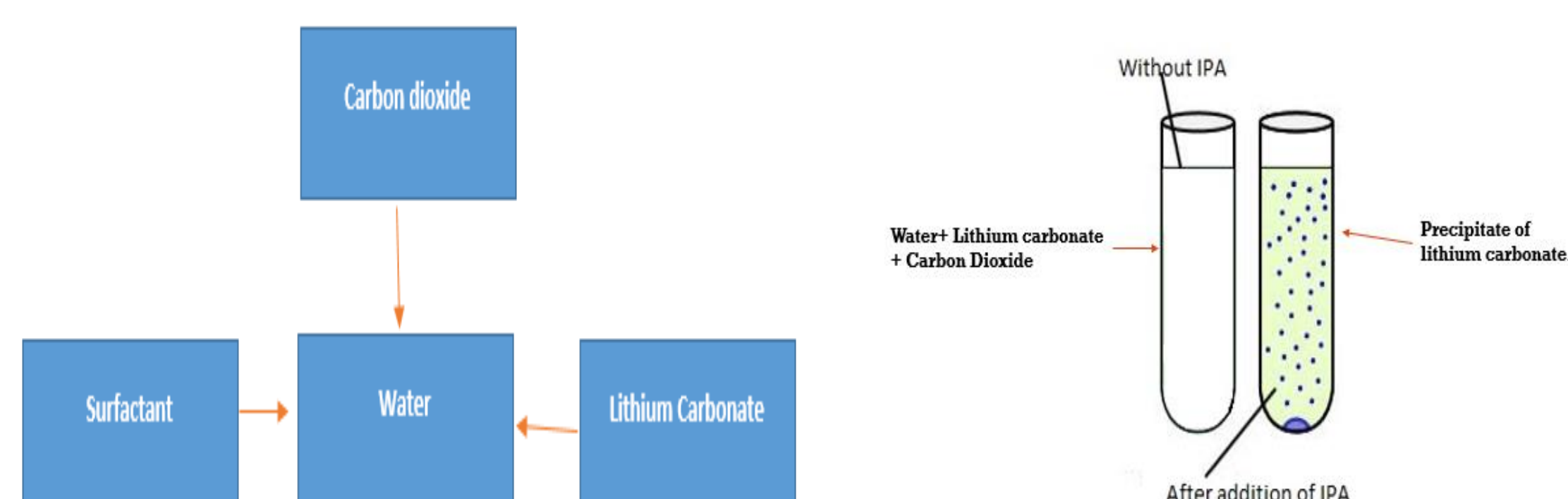
1. Microfluidic reactors have the following advantages: small volume, large surface area, control over reaction and efficient mixing¹.
2. For this study, CHEMTOR fiber reactor was used² which operates as a massively arrayed microfluidic environment and can be industrially scaled for high throughput.

MATERIALS

1. Lithium carbonate - Solute
2. Carbonated water - Solvent
3. Isopropyl alcohol - Anti-solvent (precipitating solution)
4. Tricosaeethylene glycol dodecyl ether - Surfactant

METHODS

1. Principle : Dissolve lithium carbonate in water by converting it to a metastable state – lithium bicarbonate.
2. Reaction:
3. The lithium carbonate in the solution was precipitated by using isopropanol. This technique is called fractional precipitation.
4. Process: $\text{Li}_2\text{CO}_3 + \text{CO}_2 + \text{H}_2\text{O} \rightleftharpoons 2\text{LiHCO}_3$



5. The effect of temperature, collection reservoir (dilution), surfactant concentration and flowrate has been studied

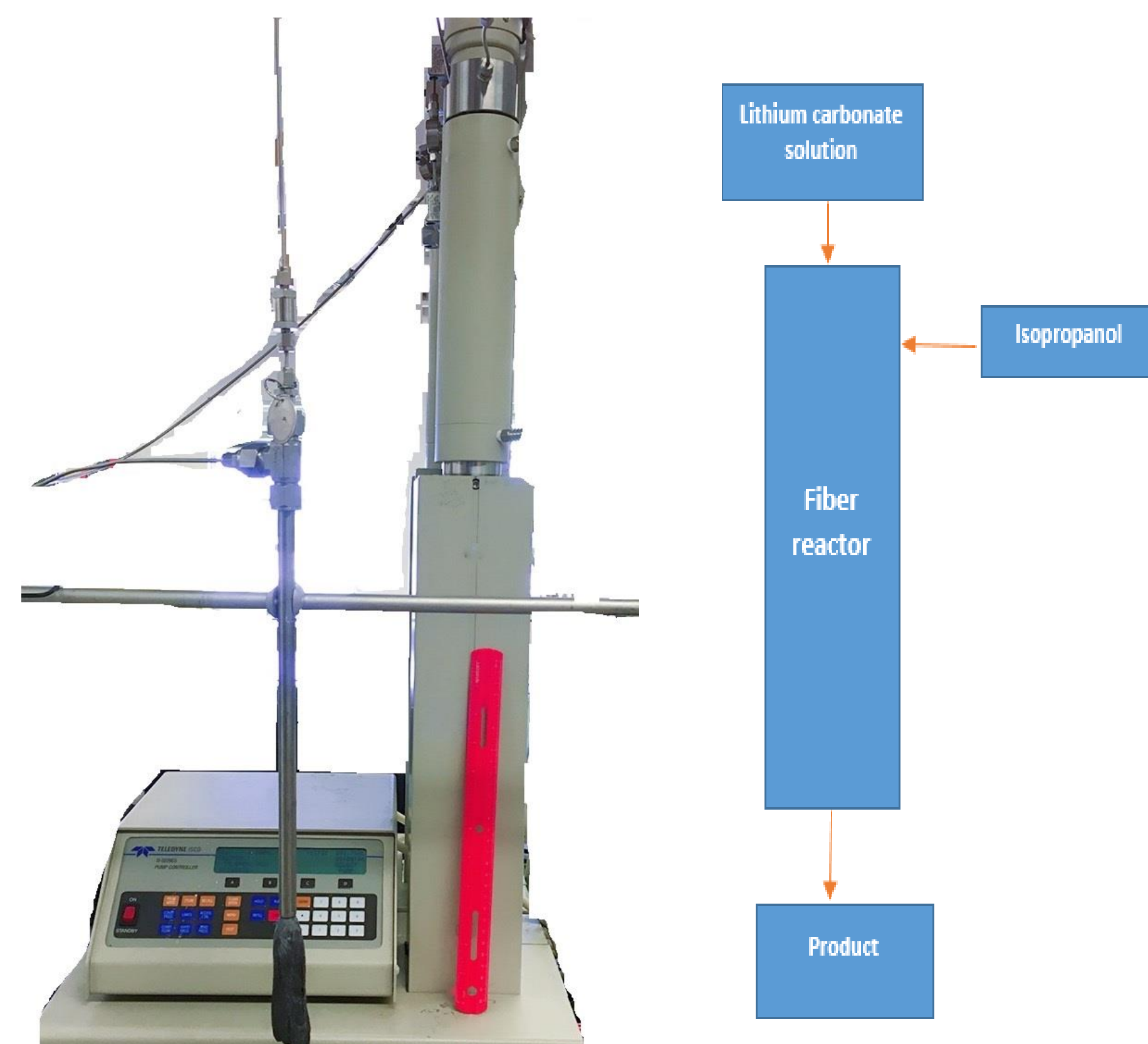


Fig: Reactor Setup

Fig: Schematic of process in the Reactor Setup

REACTOR SPECIFICATIONS

1. Stainless steel fibers.
2. Stainless steel tubing
3. 2,40,000 fibers
4. Diameter of fiber: 12 microns.
5. Inlets : 2
6. Outlet : 1
7. Flow in the channels is defined by the fibers and ISCO syringe pumps.

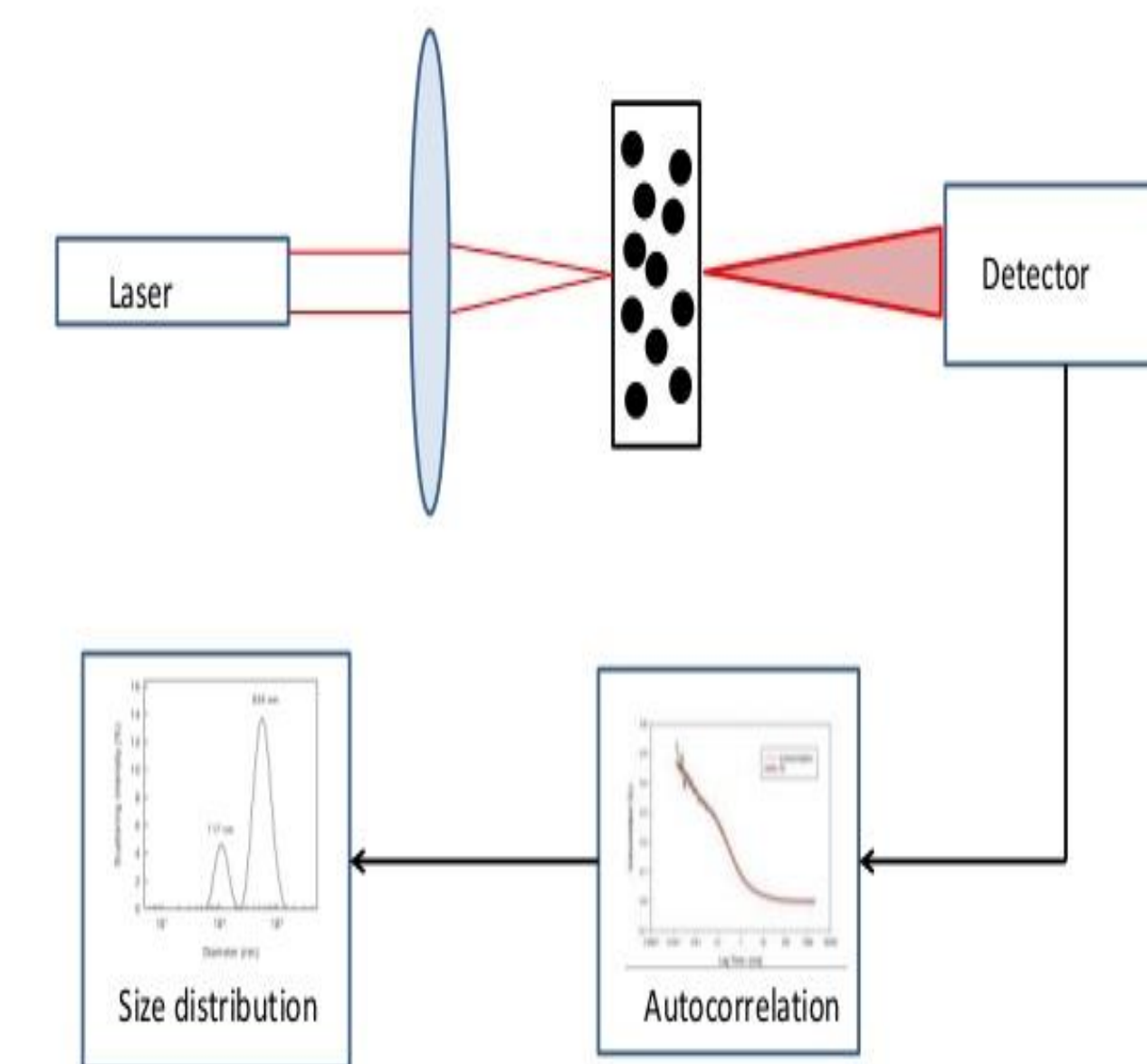
ANALYSIS

1. The size of the particles in dispersed in liquid were analysed using 'dynamic light scattering' (DLS) technique.
2. Size of the particles were given by intensity plots and Pdl (polydispersity index)
3. Intensity plots tell about the size distribution i.e. if particles of multiple sizes are present .
4. Poly dispersity index (Pdl) gives an estimate of particle size distribution
5. Usually measured on a scale of 0-1 and is given by

$$Pdl = \left(\frac{\sigma}{d}\right)^2$$

where, σ = standard deviation and d = average diameter.

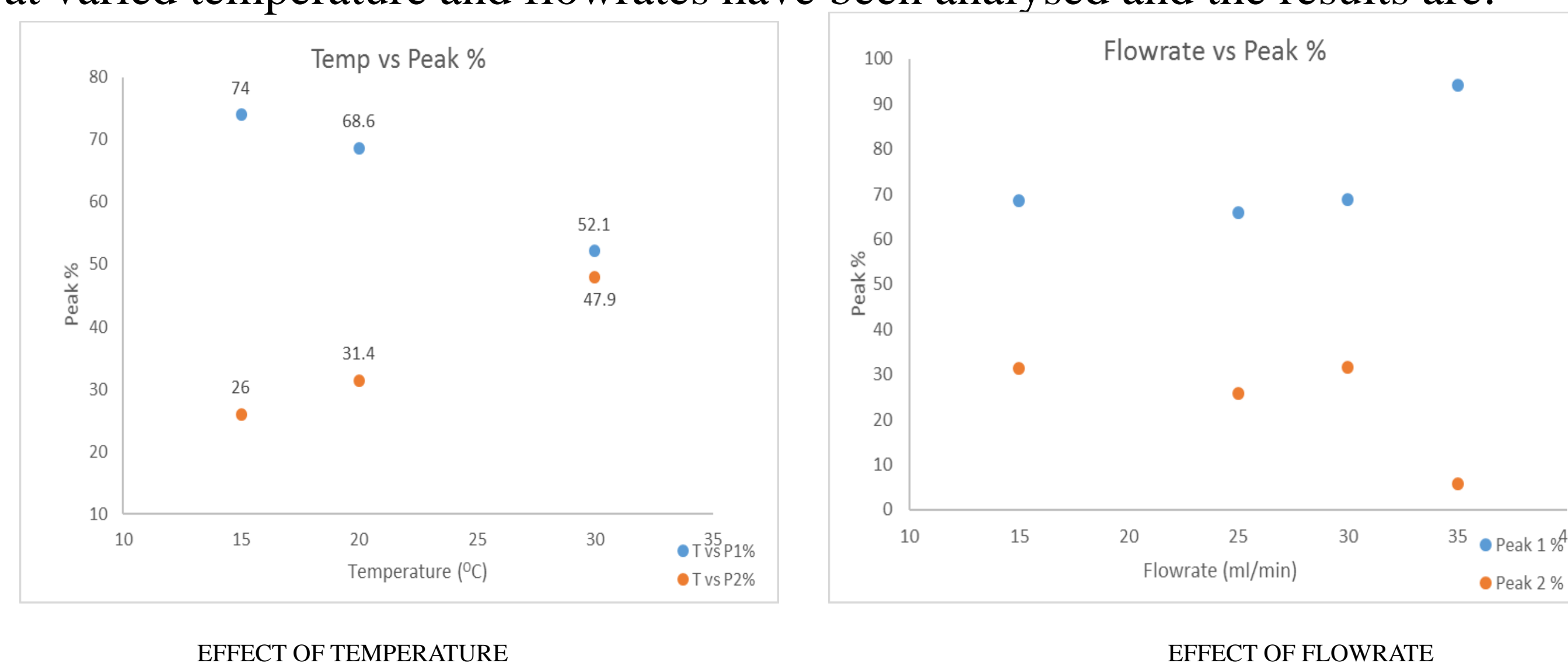
DLS – how it works



<https://img-cdn.gettyimages.com/photos/1502707604?i=1&app=691151&atomic-light-scattering=4-438.jpg?h=1432&w=1024>

RESULTS and DISCUSSIONS

Samples produced from the fiber reactor with uniform surfactant concentration at varied temperature and flowrates have been analysed and the results are:



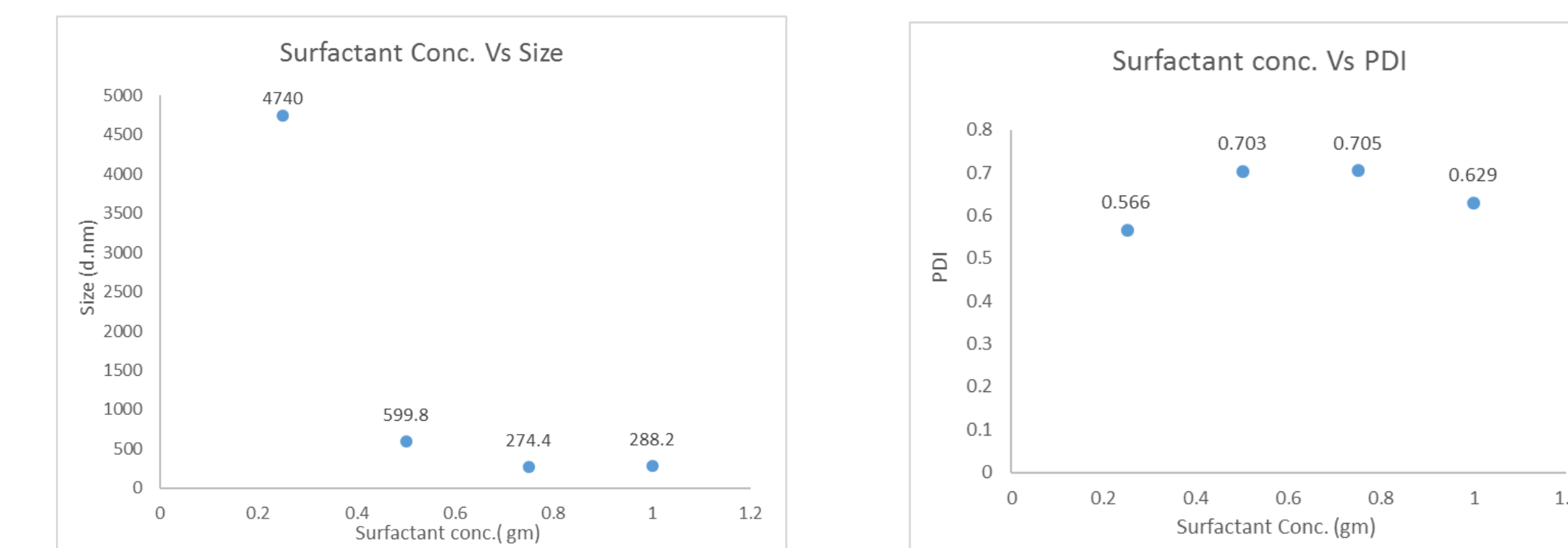
EFFECT OF TEMPERATURE

EFFECT OF FLOWRATE

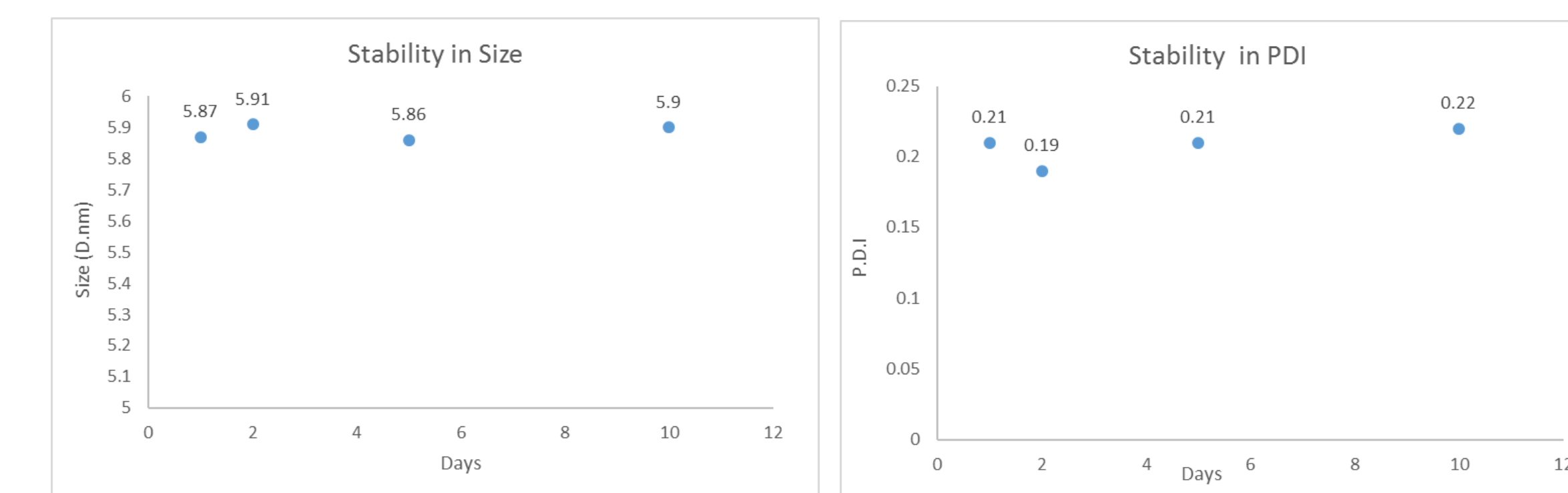
1. To achieve monodispersity i.e. Pdl<0.2, intensity should be a single peak.
2. Lower temperatures and optimal flowrate of 35ml/min were seen as favourable for this process.

EFFECT OF SURFACTANT

Samples produced at T=20°C and 15ml/min with varied surfactant concentration.



1. Increased surfactant concentration resulted in lower average particle size and Pdl has increased
2. Addition of surfactant to collection reservoir has resulted in stable product.



CONCLUSIONS

1. Synthesis in fiber reactor has resulted in smaller particles when compared to traditional batch process.

Process	Size (d.nm)	PDI
Batch	839	0.630
FR	490.8	0.471
FR (with surfactant)	5.612	0.218

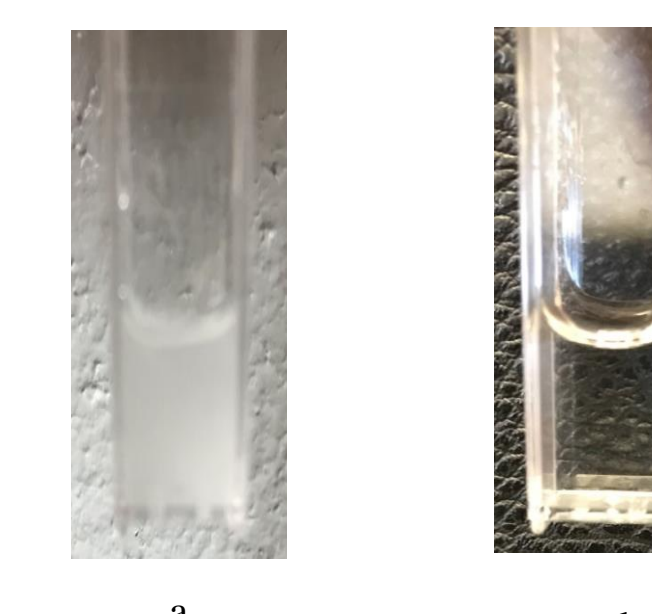


Fig : Size and Pdl of particles produced in batch vs fiber reactor

Fig : Samples synthesized in a)batch vs b)fiber reactor

2. Formation of nanoparticles was greatly affected by process temperature, flowrate and surfactant.

FUTURE WORK

1. Obtain SEM images of the particles to observe particle growth
2. Work towards achieving high throughput.

REFERENCES

1. Zhao, Chun-Xia, et al. "Nanoparticle synthesis. in micro-reactors." Chemical Engineering Science 66.7 (2011): 1463-1479.
2. Massingill, et al. "High efficiency non-dispersive reactor for two phase reactions."
3. Wang, Jee-Ching, et al. "On one dimensional self-assembly of surfactant coated nanoparticles." The journal of chemical physics (2006)
4. Rice, C, et al. "Impact of anode catalyst layer porosity on the performance of a direct formic acid fuel cell." Electrochemical Acta 62 (2012)