Hydrodynamic Cavitation assisted Emulsification and its utilization in manufacturing of Acrylate Copolymer Nano-Emulsion: A review.

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Abstract: Traditionally, nano-emulsions are manufactured using semi-continuous methods (mechanical agitation and acoustic cavitation) that requires lot of energy. Because of its properties like stability, translucent appearance, small sized droplet and easily adjustable rheology, nano-emulsions are widely used in the coatings industry. Industries are increasingly moving towards greener manufacturing technology in order to improve the product quality and reduce production costs. Cavitation-based technologies are gaining interest among processing technologies because they are cost-effective in operation and are able in comparison to the conventional methods to obtain superior processed products. In addition, following recent developments in cavitation technology, wide-scale application for the processing is referred to be possible only in case of Hydrodynamic Cavitation method. In bio-fuel synthesis, extraction of useful products, food processing, emulsification and waste remediation, the Hydrodynamic Cavitation has shown its usefulness. This review includes a general overview of Hydrodynamic Cavitation based emulsion polymerization. Conventional manufacturing process for poly (Styrene/2-Ethylhexyl Acrylate) copolymer nano-emulsion and emulsification using Hydrodynamic Cavitation is reviewed in detail. Synthesis of poly (Styrene/2-Ethylhexyl Acrylate) copolymer nano-emulsion using Hydrodynamic Cavitation based emulsion polymerization is proposed. Furthermore, future paths are suggested after thorough consideration for practical viability.

Keywords **–** Hydrodynamic Cavitation, Emulsification, Nano-Emulsion, 2-Ethylhexyl Acrylate, Venturi, Orifice Plate.

1. Introduction:

Increasing demand in the specialty chemical sector has driven the industries to transform their current technologies for efficient alternatives. Research is being carried out in the field of greener processing technologies and numerous alternatives have surfaced; out of these alternatives Hydrodynamic Cavitation has shown greatest potential. Hydrodynamic Cavitation is a state-of-the-art technology that has a wide array of applications ranging from water purification, food processing, nanomaterial synthesis and extraction to production of green fuels such as biodiesel, methanol and ethanol [1], [2].

Nano-emulsions are liquid in liquid dispersions having particle size ranging from 50 to 100 nm and are characterized by kinetic stability. Small particle size of nano-emulsions helps to achieve improvement in properties such as optical transparency, stability, high surface area per unit volume and tunable rheology. Major components of nano-emulsion are oil, water and an emulsifier [3]. Nano-emulsions are stable for longer durations; stability ranging from few months to years. Acrylate copolymer nano-emulsions are already an important part of acrylic waterborne coatings. Acrylic waterborne coatings are widely used in decorative coatings, industrial coatings and building coatings [4]. Acrylic coatings are another form of organic coating with a wide range of applications. This group of resins includes polymers and copolymer blends of polyethylhexyl acrylate and polymethyl methacrylate. Acrylics are a type of resin that are fast setting and are easily pigmented. They have high gloss, good color retention, and UV resistance. They are weather-resistant, but not generally acid- and alkali-resistant. Acrylics are mostly used in coatings for protection against outdoor exposure because of these properties [5].

Design and synthesis of emulsions with droplet sizes potentially in the nano and submicron range is a critical requirement in many manufacturing processes, despite the fact that the process is extremely energy intensive. Droplet size distribution is an important parameter as it defines the properties and activity of emulsions. The applications in the food and pharmaceutical industries are governed by droplet size distribution as well as stability of emulsions. Oil-in-water emulsions also have considerable potential in petroleum, polymer processing and cosmetic applications, in addition to food and pharmaceutical applications. Lower size (micro to nano) emulsions can be generally produced with the help of ultrasonic generators, high energy stator-rotor systems, and high-pressure homogenization, all of which are energy intensive, resulting in significantly increased processing costs [6], [7]. In the field of emulsification, Hydrodynamic Cavitation process boasts superior product quality, excellent scalability, lower energy consumption, continuous process ability and minimized process time over its conventional counterparts such as high pressure homogenization, ultra-sonication, phase inversion temperature and emulsion inversion point [1], [7], [8], [9].

In general, Hydrodynamic Cavitation exhibits great potential in the field of emulsification. Hydrodynamic Cavitation can provide greener processing methods while demonstrating efficient utilization of energy and resources. I have extensively reviewed the application of Hydrodynamic Cavitation for manufacturing of Acrylate Copolymer Nano-emulsion- poly (Styrene/2-Ethylhexyl Acrylate) and this review contains detailed information on Hydrodynamic Cavitation assisted emulsification as well as discussion on necessary parameters to be evaluated for executing this process effectively.

2. Hydrodynamic Cavitation – Principles:

Hydrodynamic Cavitation is based on the passage of liquid through a constriction like a venturi or an orifice, a winding or throttled valve, leading to a fluid velocity increase to the detriment of local pressures and a pressure drop in vena contracta below the threshold pressure that encourages cavity formation. The resulting implosion of cavities occurs as the pressure recovers and the liquid jets expand downstream of the constriction [10]. Temperatures of up to 10,000 K, as well as high-pressure shockwaves and free radicals,

are produced by imploding cavities, causing changes in the physical and chemical properties of the process fluids [11]. A storage tank, pressure gauge, pump, and cavitation chamber for bubble generation make up the Hydrodynamic Cavitation system. Cavitation chamber may either be in the form of a venturi tube, an orifice or a throttling valve.

In Hydrodynamic Cavitation, the Cavitation number is a crucial element. In flow calculations, the Cavitation number (C_V) is a dimensionless number. It characterizes the flow's ability to cavitate by expressing the relationship between the difference of a local absolute pressure from the vapor pressure and the kinetic energy per volume.

It is defined as:

$$Cv = \frac{P_2 - p}{\frac{1}{2}\rho v^2}$$

Where,
C_V = Cavitation Number.
P_2 = Recovered downstream pressure (N/m^2).
p = Saturated Vapour Pressure of fluid (N/m^2).
ρ = Density of fluid at fluid temperature (kg/m^3).
v = Average velocity of fluid at constriction (m/s).

Increase in inlet pressure leads to increase in volumetric flow rate as well as increase in velocity of the process fluid at the constriction which results in a decline in Cavitation number. Cavitation occurs more often when $C_V \leq 1$, but cavities may also develop when $C_V > 1$ due to the presence of dissolved gases and other solid particles in the liquid [12]. The cavitational yield or the strength of a cavity collapse is determined by the geometry of the cavitating system. Changes in geometrical parameters such as throat area, throat perimeter, size and shape of the throat, geometrical structure (venturi or orifice), and convergent and divergent angle of the venturi can affect the number of cavities formed, cavity residence time in the low-pressure zone, and cavity collapse pressure [13]. Many researchers have stated that for higher cavitational yield, the geometrical parameter "α", which is the ratio of throat perimeter to throat cross-sectional area, should be held high. The "α" may be increased by adjusting the shape of the throat, such as using rectangular or elliptical shapes instead of circular ones, or by increasing the number of smaller holes [14]. It has also been documented that venturi-based Hydrodynamic Cavitation devices have a higher cavitational yield than orifice plates, since venturies have ability to smoothly recover downstream pressure, they create more cavities and enable cavities to expand to their full size until collapsing. Venturies generate stable cavitation and are mostly used for chemical transformations including wastewater treatment. Orifice-based Hydrodynamic Cavitation systems, on the other hand, create transient cavities as a result of downstream pressure recovery, and are thus primarily used for applications requiring high shear, such as emulsification, particle size reduction, and mass transfer-driven chemical reactions [13], [14]. The schematic representation of Hydrodynamic Cavitation set-up is shown in **Figure 1**.

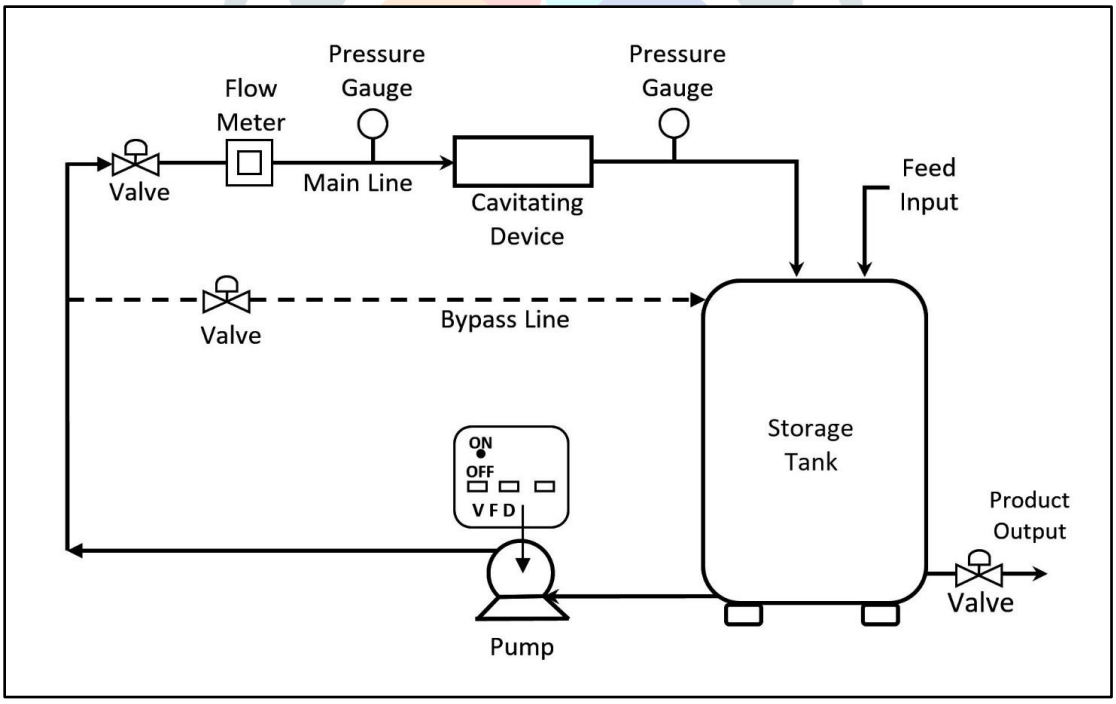

Figure 1: Schematic representation of Hydrodynamic Cavitation Set-up.

3. Conventional Emulsion Polymerization Process:

Emulsion polymerization of acrylate copolymers involves two major steps; first being the Pre-emulsification step and second one is Polymerization step. Overall process is well represented by El-Wahab et al. Int. J. Nanoparticles. Nanotech. 2019, 5:022 [3] and is summarized as follows:

Chemicals: De-ionized water, 2-Ethylhexyl acrylate, Styrene, Acrylamide, Acrylic acid, Texapone, Desponil LDPS 25, Potassium persulfate, Tergitol NP30, Ammonia solution, Sodium acetate and Biocide.

Table 1: The recipe for poly (Styrene/2-Ethylhexyl Acrylate) latex.

Sr. No.	Raw Materials	Function	Weight%
Initial Reactor Charge			
1.	De-ionized Water	Dispersion Medium	37.2
2.	Tergitol NP30	Non-ionic Emulsifier	0.4
3.	Sodium Acetate	pH Stabilizer	0.1
Monomer Mixture (4 h feed)			
4.	Styrene	Monomer	31.6
5.	2-Ethylhexyl Acrylate	Monomer	18.4
6.	Acrylic Acid	Monomer	2.0
7.	Acrylamide	Monomer	0.5
8.	Desponil LDPS 25	Anionic Surfactant	2.5
9.	Texapone	Anionic Surfactant	0.5
Initiator Mixture (4 h feed)			
10.	Potassium persulfate	Initiator	0.3
11.	De-ionized water	Dispersion Medium	6.5
Specifications			**Value**
12.	Solid Content %		53
13.	Gravimetric Conversion %		97.5

Major Processes are summarized below:

Preparation of Pre-emulsion: Pre-emulsion process is carried out by using high speed homogenizer (Ultra-Turrax). De-ionized water, ionic surfactants: Desponil LDPS 25, Texapone, Monomers: Styrene, 2-Ethylhexyl acrylate are added to reaction vessel and homogenized for 30 min. While homogenization is being carried out, specialty monomers: Acrylic acid and Acrylamide are added continuously in reaction vessel. This process is carried out at 25 °C.

Polymerization Process: Polymerization apparatus consists of a stainless steel reactor equipped with thermometer, reflux condenser and mechanical stirrer. Provision is made for purging nitrogen through the reactor during polymerization. This process is carried out at 80 °C.

Step-wise procedure for synthesis of poly (Styrene/2-Ethylhexyl acrylate) nano-emulsion is as follows:

1. Pre-emulsion mixture of styrene, 2-Ethylhexyl acrylate, acrylic acid and acrylamide is emulsified by anionic surfactants (Desponil LDPS 25, Texapone) in small quantity of deionized water and homogenized at 1000 rpm for 30 min.
2. Dissolve Potassium persulfate initiator in a small quantity of de-ionized water.
3. Charge the reactor with de-ionized water, non-ionic surfactant Tergitol NP30, pH stabilizer; add 5% of pre-emulsion mixture to the reactor gradually over a 15 min time interval. During this process, the speed of agitator is maintained at 100 rpm and temperature is maintained at 80 °C. Micelle formation is allowed for additional 15 min.
4. Further, remaining pre-emulsion is added to the reactor through dropping funnel gradually over a 175 min time interval.
5. While carrying out steps 3 and 4, the initiator solution is added continuously in a dropwise manner.
6. Once addition of all ingredients is complete, polymerization is carried out for 3 h and the reactor is held at 80 °C for additional 1 h to remove any free and unreacted monomer.
7. Reactor is cooled to 30 °C and ammonia solution is added for adjusting pH to 8. Further biocide is added to the latex and material is filtered through a 100 mesh filter.

4. Emulsification using Hydrodynamic Cavitation:

The process of dispersing a liquid into another immiscible liquid, known as emulsification, is an important step in the cosmetic, food, pharmaceutical, and nutraceutical industries. The emulsification process using cavitation is aided by the breakdown of bubbles at the liquid–liquid boundary, which lowers interfacial tension and breaks the interface, allowing droplets to disperse more easily in the continuous phase. Furthermore, the emulsion formation mechanism could occur through the collapse of a cavity in a less viscous oil phase that produces high micro turbulence and increases the interfacial area between two immiscible phases [15]. Mechanism of emulsification using Hydrodynamic Cavitation is illustrated in **Figure 2**. Surfactants, in addition to the shear forces used in the Hydrodynamic Cavitation process to minimize emulsion size, have an effect on emulsion formation by lowering the interfacial tension between two immiscible stages, resulting in smaller droplets. The optimal use of surfactants demonstrated a strong packing strength that preserved the emulsion system's hydrophobicity and hydrophilicity under cavitation conditions [16]. As a result, the surfactants' steric hindrance retains the repulsion between the droplets, while cavitation effects fragment the droplets and regulate the size distribution depending on the amount of shear applied. Overall, the combined impact of surfactant and Hydrodynamic Cavitation can not only decrease the emulsion size but also improve emulsion stability [13].

Nano-emulsions, as compared to macro-emulsions, have a special significance in food, pharmaceuticals, cosmetics, and paints that require a particular amount of energy to achieve the desired droplet size. Mechanical agitation (homogenizer, fluidizer, colloid mills), acoustic cavitation (ultrasound processor) and equivalent conventional high-energy methods have been reported to be inefficient for large-scale processing due to their low energy efficiency [13]. Besides that, low-energy approaches, such as membrane emulsification and microchannel emulsification, have drawbacks such as low dispersed phase flux, poor control over droplet size, and higher fabrication and operating costs. In this regard, Hydrodynamic Cavitation has been proposed as a better option for producing nano-emulsion with the desired droplet size even on a large scale.

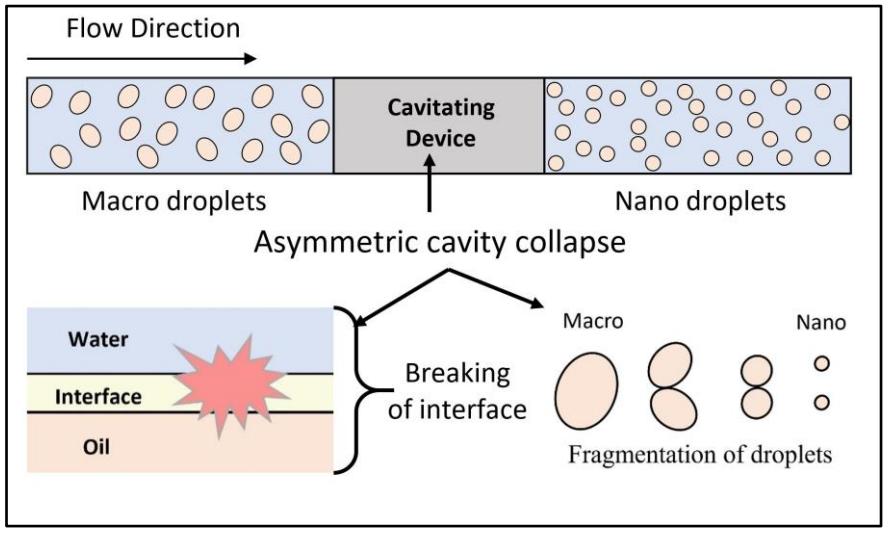

Figure 2: Emulsification using Hydrodynamic Cavitation.

5. Emulsion Polymerization of Acrylate Copolymer- poly (Styrene/2-Ethylhexyl acrylate) using Hydrodynamic Cavitation:

Schematic representation of emulsion polymerization using Hydrodynamic Cavitation is as shown in **Figure 3**. Emulsion polymerization using Hydrodynamic Cavitation will follow the same major steps of Pre-emulsification and Polymerization as of conventional approach with change in process equipment and parameters. Pre-emulsification process can be carried out by charging process vessel with de-ionized water, ionic surfactants: Desponil LDPS 25, Texapone; Monomers: Styrene, 2-Ethylhexyl acrylate, Acrylic Acid, Acrylamide, and homogenized using Hydrodynamic Cavitation.

Polymerization can be carried out in same apparatus of Hydrodynamic Cavitation; proposed step wise procedure of overall process is described as follows:

1. De-ionized water, Desponil LDPS 25, Texapone, Styrene, 2-Ethylhexyl acrylate, Acrylic Acid and Acrylamide is charged to process vessel and homogenized using hydrodynamic cavitation. This will lead to formation of a pre-emulsion mixture.
2. Once the pre-emulsion mixture is ready, pump this pre-emulsion mixture to a dedicated pre-emulsion storage tank.
3. Dissolve Potassium persulfate initiator in small quantity of de-ionized water which is stored in the Initiator storage tank.
4. Further for the polymerization process, charge the process vessel with de-ionized water, Tergitol NP30, pH stabilizer and 5% of pre-emulsion mixture and using Hydrodynamic Cavitation, start circulating feed mix through the cavitation set-up. Micelle formation will take place during this step.
5. Add remaining pre-emulsion mixture into process vessel gradually.
6. During step 4 and 5, continuously add initiator solution at initiator inlet point before Cavitating device.
7. Once all ingredients are added polymerization using hydrodynamic cavitation is carried out.
8. Reactor is cooled to 30 °C and ammonia solution is added for adjusting pH to 8. Further biocide is added to the latex and material is filtered through a 100 mesh filter.

6. Discussion:

6.1. Selection of Cavitating Device:

Vast research in the field of Hydrodynamic Cavitation has revealed availability of many different types of cavitating devices and their suitability for different processes. Widely used cavitating devices are circular venturi, slit venturi and different types of orifices out of which orifices having ability to provide high shear rates are well suited for emulsification and particle size reduction which helps in formation of stable nano-emulsions [13], [14], [17]. Effectiveness of any particular cavitating device for emulsification can be determined by measuring the smallest droplet size achieved. It is evident from research carried out by Carpenter, J et al., [13] smallest particle size achieved for O/W: mustard oil/ water emulsion is 87 nm using slit venturi. Similarly, Zhang, Z et al., [9] demonstrated that the smallest droplet size of 27 nm was achieved using orifice for O/W: Soybean/heptane/Castor/Water emulsion. The geometric parameter "β" which is defined as ratio of flow area of cavitating device to cross sectional area of pipe alters the frequency of turbulence and also have an effect on the magnitude of turbulent pressure fluctuations inside the cavitating device [1], [18], [19]. As a result, it is preferable to use a cavitating system with a higher value of "β" since a lower value will not only result in a higher pressure drop across the cavitating device but also increase the amount of energy for pumping to achieve required Cavitation number [13]. Furthermore, when Cavitation number "C_V" is low, lower values of "β" cause liquid to flash which leads to formation of cavity cloud and reduces the cavitational intensity [20], [21].

6.2. Time and Temperature for two major steps:

Time and temperature requirements for conventional pre-emulsification process are 30 min and 25 °C respectively. Similarly, time and temperature requirements for pre-emulsification using Hydrodynamic Cavitation should be determined experimentally and factors affecting time and temperature of pre-emulsification are quantity of overall pre-emulsion to be processed and accumulated process fluid in the system. Temperature gain is directly proportional to time for which the cavitation takes place and greater time for homogenization may require cooling of process fluid. In conventional approach, time required for overall polymerization is about 7 hours and polymerization proceeds at 80 °C. Localized temperature in Cavitating device at molecular level can go as high as 10,000 K making polymerization possible at cavitating device itself without the need for bulk heating and maintaining whole process fluid at 80 °C. Hence evaluation of time and temperature requirements for both major processes of emulsion polymerization using Hydrodynamic Cavitation is necessary.

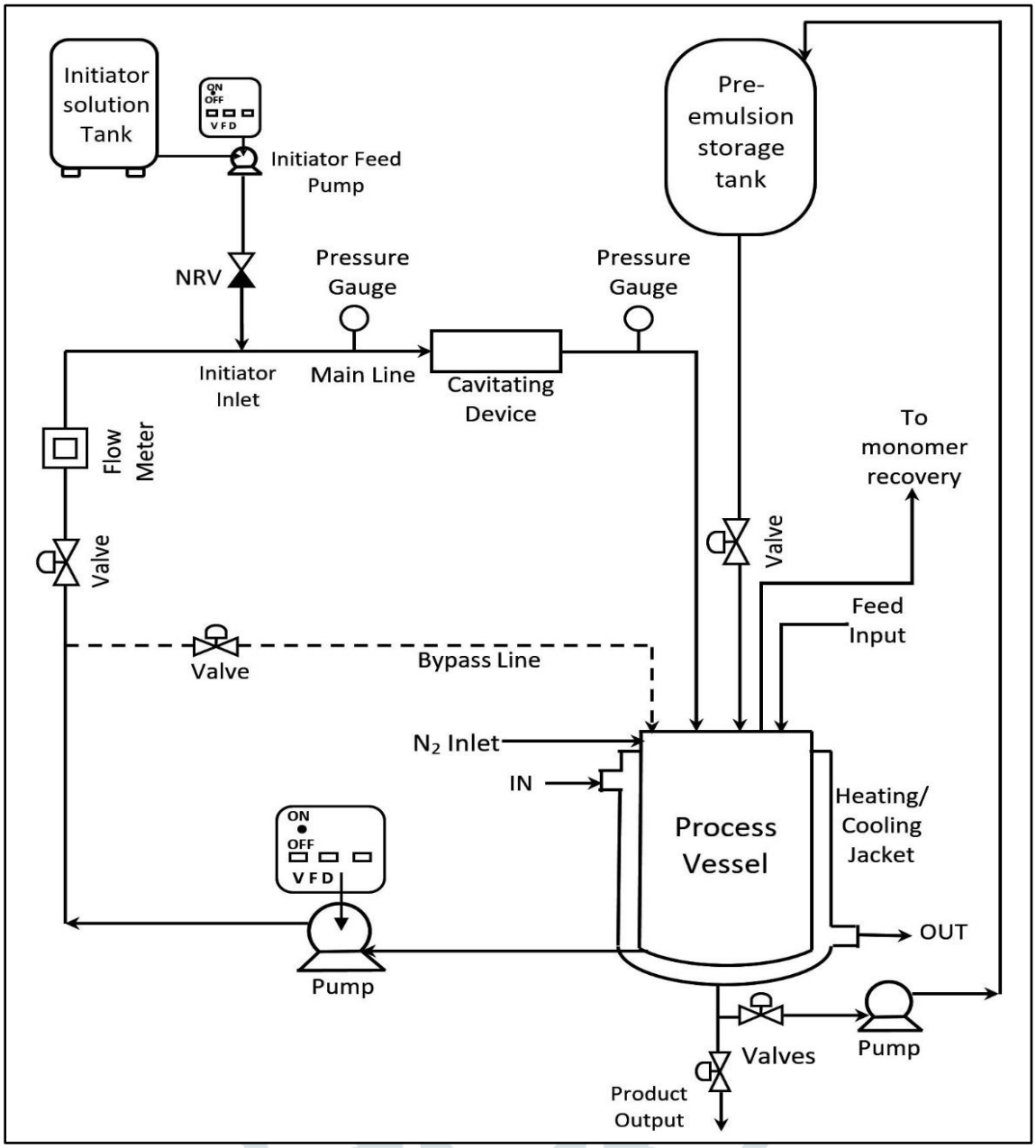

Figure 3: Schematic representation of Emulsion Polymerization of poly (Styrene/2-Ethylhexyl acrylate) using Hydrodynamic Cavitation.

6.3. Fluid level in process vessel:

It is important to make sure that a specific level of process fluid during both major steps is maintained inside the process vessel. If this minimum level of process fluid inside the process vessel is not maintained, there is risk of air entering into the process line which can lead to system failure and risk of flashing with explosion hazards. This minimum level is directly proportional to the amount of process fluid accumulated in the Cavitating Line. Special arrangements are to be made so that the outlet of the main line from the cavitating device is submerged well below the level of process fluid inside the process vessel; this will reduce or eliminate foaming caused by pressurized process fluid entering into the process vessel. Therefore arrangements should be made to monitor and maintain this process fluid level. Special design modifications, like adjusting angle of main line outlet into process vessel can be made to increase mixing inside the process vessel by harnessing the pressure by which process fluid is discharged into process vessel; this can also be done by adjusting the inlet position of process fluid into the main line which will modify the suction pattern. The schematic representation of processes vessel with necessary arrangements is shown in **Figure 4**.

6.4. Cavitating parameters for two major steps:

Cavitation inside cavitating device depends on Cavitation number "C_V", geometrical parameters "α" (ratio of throat perimeter to throat cross-sectional area) and "β" (flow area of a cavitating device to cross sectional area of pipe). The Cavitation number in turn depends upon the average velocity of fluid through the constriction, saturated vapor pressure of fluid and density of fluid. Geometrical parameter "α" will decide the cavitational yield and "β" will control the turbulence inside the cavitating device. Pre-emulsification step involves highly flammable materials and along with "β", overall vapor pressure of the pre-emulsion mixture should be considered while determining inlet pressure which will decide average velocity of fluid through constriction. This should be critically evaluated to avoid flashing of processing fluid while it passes through cavitating device. Similarly, integrating above information, suitable processing conditions for the polymerization step should be determined along with evaluation of suitable geometrical parameters "α" and "β" so that it leads to stable copolymer nano-emulsion.

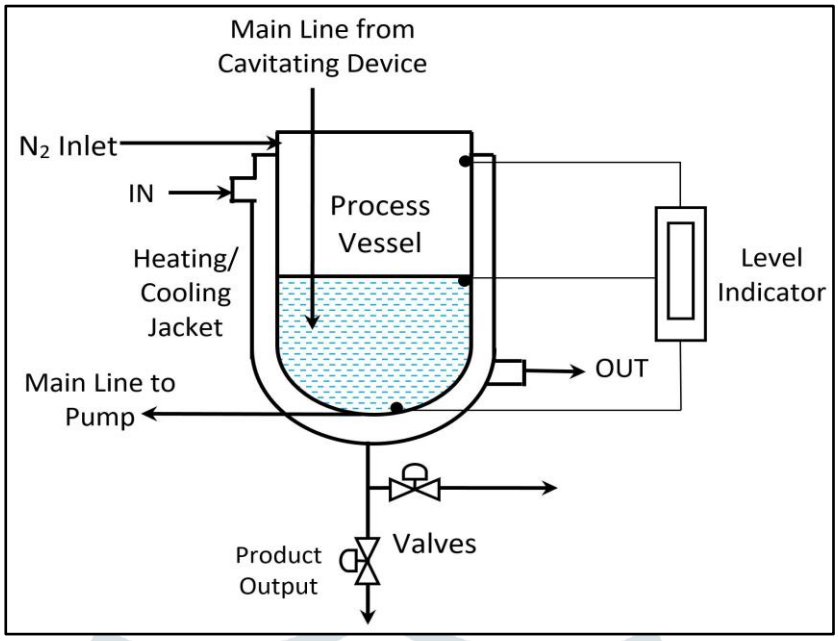

Figure 4: Process vessel with level indicator and main line outlet below fluid level.

7. Conclusion:

Increase in the demand of specialty paints has resulted in increase in research in the field of nano-emulsion development as well as optimization of manufacturing processes. Acrylate copolymer nano-emulsions which serve as binder to waterborne coatings helps to develop novel paint systems with superior gloss, weather resistance and excellent stability. Hydrodynamic Cavitation helps us to achieve above mentioned properties in a more efficient way as compared to conventional methods. This review demonstrated that Hydrodynamic Cavitation can be used in manufacturing of poly (Styrene/2-Ethylhexyl acrylate) copolymer nano-emulsion by effectively harnessing the effect of bubble implosion. Choosing an appropriate Hydrodynamic Cavitation reactor configuration with a suitable constriction type can have a significant impact on the overall processing mechanism. Furthermore, critical optimum conditions such as reaction solution temperature, inlet pressure, and Cavitation number must be considered for the best possible results. Stepwise procedure, schematic process flow diagram and critical process parameters for manufacturing of poly (Styrene/2-Ethylhexyl acrylate) copolymer nano-emulsion by using Hydrodynamic Cavitation are discussed. Future investigation should focus on execution of the aforementioned manufacturing process using Hydrodynamic Cavitation by making necessary amendments and determining critical operating conditions. To summarize, Hydrodynamic Cavitation can be effectively used as a greener processing technology thus replacing the conventional techniques used for emulsion polymerization and has limitless scope in the field of nano-material synthesis.

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