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A Study on Base Processes for Selected Reactions of Microreactors

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Abstract – For a variety of chemical reactions Microreactors have outstanding mass and heat transfer efficiency. In terms of energy performance, yield, protection, reliability and scalability, the usage of micro-reactors provides also advantages over traditional reactors. In comparison to the batch phase, Microreactors dissipate heat efficiently and also very exothermal. Also if micro reactors will synthesize chemical products in limited amounts only, the amount of micro channels can be added to the industrial phase. The study discussing micro-reactors characteristics, Experimental process for nickel nanoparticles synthesis in Microreactors, Heterogeneous catalytic response Microreactors, Microreactors building, Microreactors' advantages Comparison of batch and micro-reactors Comparison various forms of reactors/devices used in chemical industries for process intensification

Keyword – Microreactors; Nanoparticles

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INTRODUCTION

The continuous reactors with internal measurements of less than 1 mm are the Microreactors. In order to increase surface efficiency, the capillary channel's diameter may be decreased to 10 to 500. In Microreactors the conversion is more than just a batch operation, since the heat and mass transport limit is decreased. Stainless steel, silicon, glass and polymers are constructed of Microreactors. Innovative steel The running of pilot plants involves micro-reactors Due to its tolerance to different solvents, acids, bases and other reagents, glass is the most used medium used for the manufacture of micro-reactors. Silicone has excellent thermal conductivity, thermal transfer capability, and is thus used in high and low temperature reactions in micro-reactors. Micro reactors may be manufactured with polymers with a poor tolerance to most reagents and solvents that are of limited performance. The micro reactors are now market-displayed, generated by several engineering companies including BTS Ehrfeld, CPC, IMM, Microglas, Microinnova and others. In the production of miniaturized instruments for chemical, synthetic uses, i.e. Microreactors, micromachining technologies are being implemented. A Microreactors is a system that contains a range of micrometers in which a number of orders of magnitude smaller than traditional batch reactors are carried out in the space of reaction. These reduction results give Microreactors a series of desirable characteristics. In this article the technology of Microreactors is presented and a Microreactors Scheme explains the efficient numbering processes between research and development by plant construction. There are many descriptions of the application for Microreactors to manufacture the process, including combining, reactions, emulsification and liquid concentration.

The word "Micro-reactors" initially used in the science of catalysis to refer to small tubular reactors, but with progress in the field of micro-reaction technology it has become associated with a multi-sub-millimeter device consisting of constant fluid and chemical reactions. The promise of these micro-reactors is less waste, lower utilities, better throughput testing and protection benefits over traditional systems. The limited quantities of reactions of the Microsystems coupled with the high heat and mass transfer rates allow for reactions that are more violent and yield more efficient than traditional reactors. In addition, the high heat transfer and easy containing of a limited quantity will safely lead to new reaction pathways which are considered too difficult to monitor in traditional macroscopic devices. The latter attribute is especially relevant when highly reactive intermediates are involved in chemical transformations. Process intensification centered on micro equipment is a new concept in chemical engineering which, by growing the size of the chemical plant, aims to reduce cost and energy, along with the environmental effect. With a decline in the scale of the machinery by many magnitudes, considerable economic advantages may be gained, improved inherent protection and reduced environmental impacts. 1 Moreover, the limited scales used limit exposure to radioactive or dangerous chemicals and, in case of a fugitive reaction, the sealed structure of the micro-reactors facilitates containment. The advantages of switching from batch to processing, using high mixing and heat transfer speeds in intense reactor technology and the possibility of versatility in a multiproduct system are investigated. The results are also discussed. One of the main difficulties of a chemical engineer is the inclination to higher added performance chemicals with improved commodity purity.

Characteristics of Microreactors

In comparison with batch processes, Microreactors have improved efficiency and several functional advantages. The limited scale of the reactors enables fewer reagents to be used under stable conditions to boost reactions. With a less residence period, lower volumes of reagents, less catalyst volume and less waste product emission, Microreactors tests may be conducted. Microreactors are lightweight in weight compared to massive reactors. They have a compact architecture, an efficient mixing, a short distance of molecular diffusion and a stronger process control with lower power usage. The small scale, large surface-to-volume ratio of microstructures offers various advantages for micro-reactors. Hydrogenation is strongly exothermic in nature, like some f reactions such as nitration. Heat reduction is also a major element in regulating these reactions. Thermo transfer is carried out very quickly across the surface of Microreactors, by taking advantage of the fact that Microreactors have a wide region per unit volume (10,000 to 50,000 m^2/m^3), This controls accurately the temperature. The residence time regulation is another valuable function of Microreactors. Residence time may be changed by changing flow speeds into a couple of seconds in Microreactors.

Experimental procedure for synthesis of nickel nanoparticles in a Microreactors

In continuous coiled micro reactors which are bound to the y-shaped junction were synthesized nickel nanoparticles. Reduced hydrazine hydrate in an aquatic with and without the capper was used for the reduction of $NiCl_2.6H_2O$ to nickel ions. During the batch phase the first sample was prepared the same. In two separate syringe pumps, solutions A and B were added. The syringes were fitted with syringe pumps. The seed was entered through a connection with Microreactors. The Microreactors were built in circular winding with a 173 cm long, 1.2 mm inner diameter copper material. A figure shows the synthesis method. The solution was stored in sample bottles and the color found in dark blue at a steady reactor temperature of 60 ^0C during tests of 1mL / min. Often, from dark blue to blue sky, the fluid changed and the component was placed at the bottom of the conical bottle. The finished product was treated with purified water many times for 30 minutes at 1500 rpm. The nanoparticles obtained were characterized to know the impact of different parameters on the development of nickel nanoparticles.

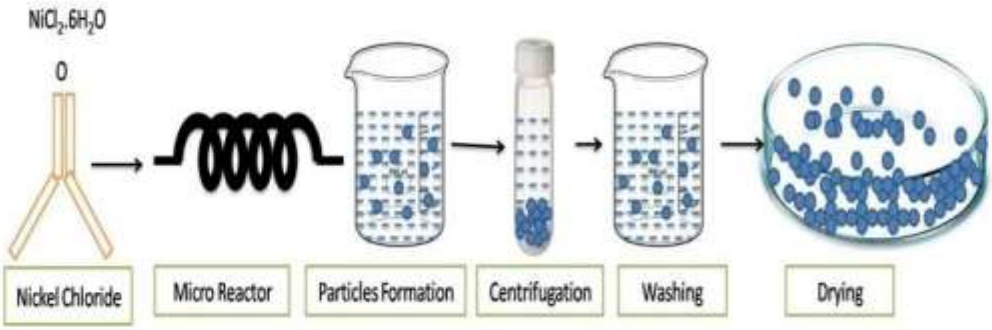

Figure: Synthesis of nickel nanoparticles in continuous Microreactors

Microreactors for Heterogeneous Catalytic Reactions

The interest in accelerated discovery of new catalysts has contributed to important progress in high-performance screening and combinatorial methods for fast screening of large arrays of catalysts. For the conversion of promising candidates into process settings, detailed characterisation of the catalyst output such as kinetics, selectivity and deactivation is required. The use of a reaktor where the density, thermal and pressure gradients are minimal simplifies these experiments, but with conventional reactor technologies, the achievement of such conditions could present a challenge. Increased surface area/to volume ratio for increased heat transmission provides a specific benefit in the micro fabrication of chemical reactors including micro-press beds for effective catalyst research like lower transport restrictions. In macroscopic test systems, the micro fabrication often provides flexible monitoring of reactor geometrics.

Usually, traditional catalyst tests of small tubular reactors are carried out. A subsequent 60 μβ Catalyst Particle nano-manufactured channel reactor shows the possibility for the use of a chemical kinetic micro reactor. However, because of the limited catalyst particle scale, this structure has significantly decreased strain. Low pressure drops and minor (differential operation) conversions generally help to achieve chemical kinetics for catalysts. In the other side, for subsequent catalyst research, it is desirable to provide as much catalyst as practicable. In the form of silicon-based, cross-flow package-beds, micro-reactors for catalyst testing may be used to resolve the evidently contradictory demands. This architecture incorporates short parallel beds into a large package bed that provides a short contact period with enough catalyst to characterize the reaction efficiency and post-reaction structure of the catalyst. Even the flow distribution through the big bed is done with 256 flawed channels which bring a far greater drop in pressure than the fall through the catalyst bed. Differences in the density of catalyst packaging have a marginal impact on the total flow delivery. The geometry of the cross-flow makes the bed isothermal and isobaric. In addition, the cross-flow geometry allows the usage of realistic flow speeds and catalyst concentrations and minimizes pressure reduction.

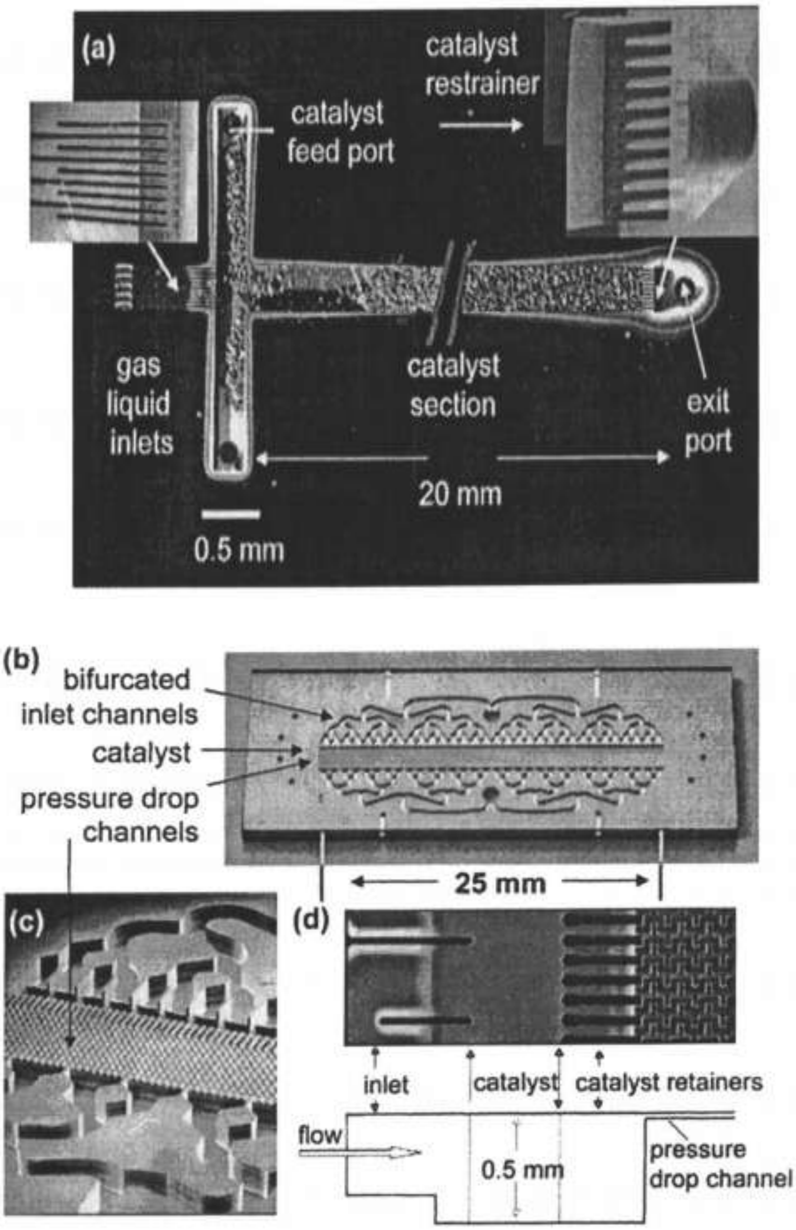

Figure: bed filled with catalyst Active carbon catalyst micro-reactors. The picture has been updated to delete the long middle portion of the catalyst so that the inlet and outlet parts can be increased (a) the following: (a) Scanning electron micrographs and press drop detail (c), cross-section in the flow-side direction. Micro-manufactured Silicium Cross-Flow Reactor – photograph (d)

Construction of Microreactors

Most developed micro fluidic fuel cells used previously accepted manufacturing methods for physical sciences and micro fluidic chips. These instruments have a micro channel, two electrodes and a lightweight liquid maintenance system. Speedy prototyping, traditional lithography of disbursement and soft lithography protocols also make micro channels classically. The channel organizations were developed in polydimethylsiloxane (PDMS) and wrapped in a strong substrate that included the conductor structure through gearing towards the electric microfluidical cell devices by side-by-side fluxes. The literature includes various channel strategies, as stated by Kjeang et al. The lenient, lithographed approach is industrialized to strengthen a silicon wafer by pretreating a substrate or by use of a photo resist sheet to apply a coating. Photoresist materials spin-coated in the layers of the microchannel which define how the layers work,

The coated substratum is softly heated on a hot plate for a stable photographic mechanism. The resolution is measured with CAD tools, including a photo mask and allowing high-resolution picture printing. It is then allowed to consume UV radiation, which creates two portions of the photorist which are exposed and unexposed. A substrate absorption fluid is used for the measurement. A decoration with a positive effect reveals a decent polymerized photo cut. PDMS is used for duplicating the configuration of the channel with moulding, accompanied by vacuum exposure on a warming tray.

Applying enzymatic bio fuel (or fuel cells in general) to organic catalysts is several microfluid Microreactors compliant. Via catalysis of macromolecules (glucose), bio fuel cells work or synthesis the power of the redox reaction using an organic catalyst. The 2014 gross revenue base of micro-fluidic instruments amounted to $1.8 billion and it is anticipated that by 2020 it would rise by $7.7 billion. The world is improving the architecture of the reactor, in which Microreactors are produced to simulate and analyze chemical reactions. The development in the industry was driven by the availability of good materials for the production of micro reactors at low costs. A small reactor in conjunction with polymers and polymer products is generated utilizing various material forms (e.g. silicon, glass, steel and ceramics). The small reactors may be built as capillary or chip forming reactors, allowing their broad use, as seen in figure, in various science fields. Taking account of the related fluid properties, capillary reactors are constructed in terms of scale and duration. Chip-based Micro reactors, in comparison, are usually constructed using micromachining, etching and lithography methodologies, often quartz, glass or even some forms of plastic.

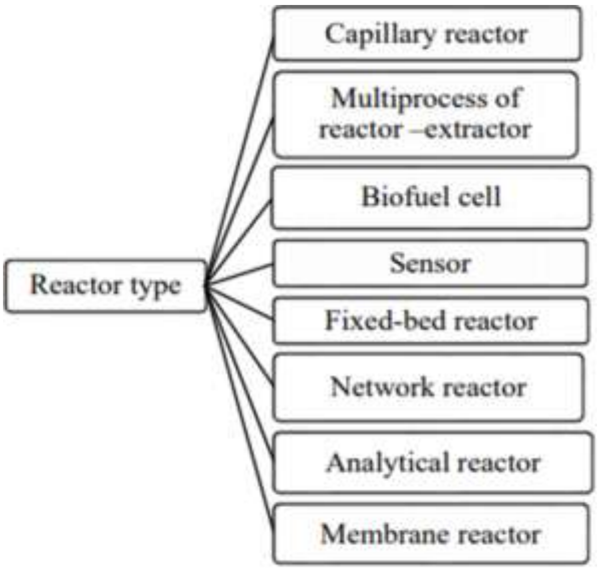

Figure: Micro reaction applications in different fields of science

Advantages of Microreactors

They have a large surface area, increased density, thermal transport and occurrence of laminar flow conditions. The benefits of Microreactors, less time of residence, narrow distribution of size is achieved in micro channels and is thus utilized in colloidal particle synthesis. The optimal conditions necessary during the synthesis reaction are: reactor mixing, short period of residence to achieve a narrow distribution of nanoparticles during the residence periods, flux conditions, pressure, diffusion rate, while the reaction takes place in limited volumes of the region of the reactor. The reactor's scale varies between 100 and 1 mm, which influences mass transfer in limited sizes, efficient thermal transfer and the surface characteristics render the reactor special. Lower risk of material generation, less fire, avoiding accidents and leakage are the advantages of small reactors. A limited volume of the reaction fluid is maintained by Microreactors with thin channel measurements that avoid mechanical failures. Thus, less reactants, accurate monitoring of process variables, compact layout and reduced power consumption are required in micro-reactors. By integrating microchannel geometry configuration and fluid flows, nano particle sizes can be accurately regulated, reproducible and polydispersed. To monitor the size and morphology, micro- and nanoscale particles may be collected. Intensifying micro-mixing and mass conversion leads to uniform nano-size delivery. There is an illustration of the benefits of Microreactors.

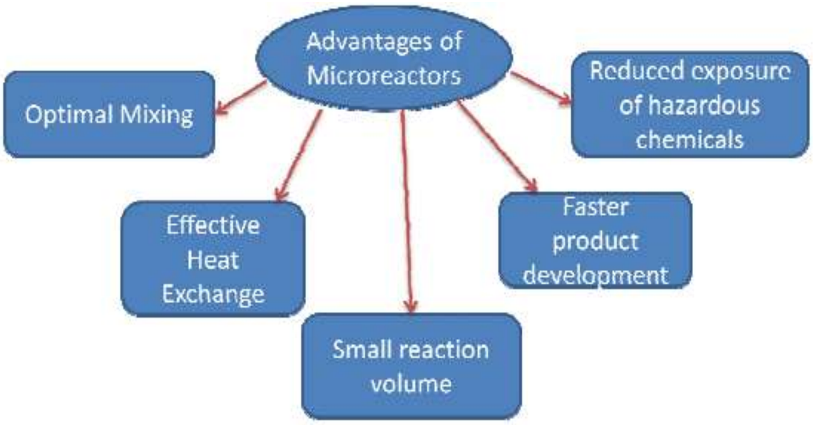

Figure: Advantages of Microreactors technology

Micro reactors have many benefits, such as high surface-to-volume, improved weight and heat transfer and strong volumetric productivity. In the Laminar flow the flow conditions prevail. As the pressure drops, the time distribution for residence and the degree of control back mixing are reduced. The cost of production is lower and the method is simple to operate. Low cost maintenance and low electricity demand. The environmental risks and protection due to limited amounts are low.

Comparison of batch process and continuous Microreactors

In a batch phase, thermal gradients and reaction are very challenging to hold away. The nucleation of nanoparticles with its development is complicated to distinguish from a batch reactor. Therefore, a combination nucleation and development lead to the polydispersal process resulting in standardized distribution of the scale. For nanoparticles synthesis, an effective mixing of the reagent is necessary. Current micro reactors have the benefit of controlling the reagents that mix together in a short amount of time. In continuous Microreactors, the required reactions can be completed in far less time. Although vast quantities of chemicals are used in batch reactors, recently, chemical compound preparation involves further machine reaction. However, several reactions in traditional batch reactors remain. This makes the operation hard to scale up. Microreactors have been designed to get out of the crisis. The mix is performed using impellers and mechanical stirring in the batch phase. Turbulence is produced as the mixing pressure rises. Uniform propagation and mixing in laminar flow situations is carried out, as is the case in Microreactors.

Different types of reactors/devices used for process intensification in Chemical Industries

The following are different kinds of flow reactors:

- **Microwave reactors:** Microwave reactors offer the reaction mass uniform local heating, which may contribute to a successful conversion of the reaction. However, for the scaling up to a continuously increasing method with higher flow rates, important research is still needed. Radial mixing and transport processes are improved through oscillating disturbed reactors by means of isolated and controlled oscillation movement. Because of this mixture form which is independent of flow rate, these reactor forms provide good stability. These reactors can be used with heterogeneous catalysts.

- **Static mixers:** The static mixer consists of moving geometric components that are specifically constructed, and enclosed inside the tubes or column, creating efficient radial mixing of two immiscible liquids through the mixer. The blended fluid is liquid, but it may also be used to combine gas sources, distribute gas into liquid or mix immiscible water. The required energy for mixing is caused by a pressure loss as fluids pass through the static mixer.

- **Membrane reactors:** Membrane reactors are integrated with one single step in reaction and membrane-based division. They may improve the conversion of balanced reactions by removing certain products through membranes. Membrane reactors reach a high reaction rate by the targeted removal from materials from a membrane of one component. However, the expense of the reactor membrane normally restricts the uses. When using heavy bases or acids, which restrict implementations, the membrane employed in these reactors is less resistant.

- **Reactive distillation:** It is a methodology that in a single device incorporates chemical reactions and product segments. This blends the reactive phase with product isolation. However, the energy intake is very strong. A major benefit of reactive distillation is the continuous removal of the substance in situ for equilibrium-controlled reactions avoiding conversion constraints.

- **Microreactors:** They are equipment that include chemical reactions with lateral measurements usual below 1 mm. By raising the surface area via the tiny characteristic dimension of the reactor, Microreactors will decrease the reaction times. Micro-reactors, though, still mean very low flow speeds.

Synthesis of Solid Particles in Microreactors

In general, the generation of solid particles is thought to be an agglomeration and plugging issue for Microreactors. However, Microreactors can be an excellent method for the continuous synthesis of particles with well-controlled properties with careful control of nucleation and expansion. This applies in particular to particles of nano-size involving several synthetic measures such as colloidal particles synthesis and corresponding surface modifications for biological and optical testing. Scale down to sub millimeter measurements in the Microreactors offers possibilities for better colloidal synthesis size and composition power. Micro-manufacturing often provides improved reactor configuration versatility, in particular the difficulty of implementing reactor-contacting schemes in macro-sized units. Single homogeneous flow for particle synthesis in Microreactors is not normally suitable since axial dispersion increases the size spread fluid movement slower close to the tube wall than at the centre variables residence periods. It is possible to reduce the dispersion effect and to achieve tiny particles by utilizing a segmented flow in which the synthesis fluid is separated into different fluid segments (captures). The segmenting fluid is used to accelerate micro-mixing by recalculating in each segment, in addition to splitting the synthesis fluid into tiny lots. Segmented gas-liquid The easy way to isolate the gas from the liquid by utilizing capillary forces is desirable for Microreactors.

The sober synthesis, a widely researched technique, of colloidal silica is a model system. The micro-reactors made of PDMS show that the water-ethanol-tetraethoxy silane mixture used in this case is chemically compatible. The Silica synthesis is characterized by fast initial development, followed by sluggish growth. In a single phase flow, synthesis is highly vulnerable to short time residence effects. Particles close to the Wall travel slowly and spend longer in the Reactor than near the middle of the Tube. The fluid is divided in the segmented flow into a series of well mixed batch reactors, resulting in a narrow distribution in duration. These findings show the value of quantifying each fluid in the microelectroplayers' residence period distribution (RTD). In the course of the measurement of the RTD in the Microreactors it is possible to introduce a means to inject the fluorescent tracer (for example, through a built-in piezoelectric bending disc element) and then to monitor fluorescence spread spatially as the tracer plug passes through the tube.

A second example of Micro-Reactors assisted particle synthesis is the synthesis of nanocristalline, semiconductor quantum points (QDs) (2-10 nm diameter), precisely CdSe. The optical characteristics and medium size of QDs in traditional batch processes are determined by variables that are difficult to monitor, such as injection phase, variations of local temperature and concentration, stirring intensity and cooling rate. A segmented flow of gas-liquid Microreactors allow fast and regulated precursor mixing. Moreover the segmented flow reduces the RTD and, hence, the size distribution of QDs generated, as is the case with colloidal silica. Silicon is used as construction material to allow nucleation and growth at temperatures up to ~300°C that are essential for good luminescence of QDs. The silicone is oxidized again in several chemical processes to provide the preferred glass surface.

CONCLUSION

Nickel nanoparticles have been synthesized by chemical reduction procedure in batch phase and in continuous Microreactors. Nickel reduction was achieved by reduction of the agent and PVP regulated the size of the particles. From the experimental work the following results were drawn: The reaction period in continuous Microreactors was 60 minutes when nickel nanoparticles were formed; the time of reaction was increased as in the batch phase. While nickel nanoparticles are reduced in the color solution of transition. In this work the spiral copper tube Microreactors (0,8 mm indoor diameter and 124 cm in tube length) and the batch process were synthesized with palladium, nickel and silver nanoparticles. The synthesis of organic compounds, biopolymers, nanoparticles and many other substances in general plays an important role in Microreactors. With complex pathways and reaction mechanisms, the tiny existence of micro-reactors can produce predictable chemical reactions. Information of processes, fundamentals, and structural materials used in the production of small reactors were provided in these works. They analyzed the impact of the different parameters, such as molar concentration, flow speeds, catalyst, temperature, residence period and their construction/manufacturing mechanisms, along with their main implementation technologies. Microstructure reactors are a highly efficient way of intensifying procedures with complex processes. Increased heat and mass transmission, stability, environmental effects, dispersed manufacturing, high-portability, remote (on- site) and technological flexibility is the advantages of using Microreactors technologies as well. In the context of specifically regulated environments, reactions in Microreactors ensure better rates and product efficiency relative to batch processes.

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