Chapter 6
A Review of Nanofluid Synthesis

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6.1 Introduction

Nanofluids are stable colloidal suspensions of nanoparticles in a chosen solvent (base fluid). The dimension of “nano-” entities is strictly defined by ISO/TS 27687 as a particle with at least one representative dimension (e.g., diameter, thickness, or length) ranging in size from 1 to 100 nm. This conventional definition of the nanoparticle is commonly agreed among nanotechnology researchers. The classification of dimension less than 100 nm arises from the phenomena that the surface area to volume ratio increases rapidly as the size of the particle is diminished. As a result, the material properties of these novel nanoparticles often deviate significantly and anomalously from their bulk (conventional) values due to the dominance of surface effects (e.g., from interfacial interactions and dominant contributions from surface energy). In other words, nanoparticles exhibit unique properties which are drastically different from their bulk characteristics. In-depth studies on the transport mechanisms responsible for these anomalous behaviors are still a burgeoning topic in contemporary literature.

The concept of nanofluid was first proposed by Choi and Eastman [1] in 1995. They observed an anomalous enhancement in the thermal conductivity when a small percentage of copper nanoparticles were dispersed in water. Subsequently, various combinations of nanoparticles and liquids have been studied for enhancing

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transport phenomena and thermophysical properties (e.g., thermal conductivity, specific heat capacity, and viscosity). Typical materials used for synthesizing nanofluids are metals (copper, aluminum, gold, etc.), inorganic oxides (iron oxide, zinc oxide, silicon dioxide, etc.), carbon-based materials (CNT, graphene, fullerene, etc.), and other ceramics (aluminum nitride, PNP, cellulose, etc.). A popular choice for solvents includes water, glycols, ionic liquids, organic liquids, and refrigerants. Other liquids explored in the literature include lubricants, oils, biofluids, emulsions, fuels (e.g., kerosene), alcohols, molten salt eutectics, etc.

Despite the vast range of materials that have been explored in the literature for synthesizing nanofluids, the general concept of dispersing nanoparticles in the base fluid is to enhance certain material properties of the base fluid for achieving enhanced performance in a chosen application. For example, high thermal conductivity is desired in the heat transfer applications (often at the expense of higher viscosity and pump penalty) while improved load-carrying capacity and non-Newtonian rheological behavior are preferred in lubrication applications (with a concomitant ability to dissipate heat rapidly). On the other hand, pump penalty is of secondary consideration in thermal energy storage (TES) applications—where the material cost ($\$/\mathrm{kWh}_t$) and enhanced specific heat capacity are of primary importance. Hence the desired fluid properties are motivated by the chosen application which in turn mediates the choice of the synthesis protocol for a specific nanofluid. Thus, it is crucial to establish a library of synthesis protocols for the select set of nanofluids for target properties (and applications).

Another important issue and common concern in nanofluids application is the long-term stability. The current architecture in nano-manufacturing is well-developed which allows us to synthesize nanoparticles with different size, shape, and structure. However, keeping nanoparticle suspended uniformly in the base fluid for long enough time is still a challenging task. Various approaches have been explored to increase the stability of suspensions. These approaches have largely yielded short-term improvement but no conclusive studies exist for ensuring long-term stability. One key idea in keeping nanoparticle suspensions stable for long time periods is to prevent them from agglomerating (which is primarily induced by Brownian motion-mediated collision and coagulation). Some of the typical strategies utilized for ensuring long-term stability will be discussed in subsequent sections.

One last major consideration in nanofluid synthesis is the cost ($\$/\mathrm{kg}$ and $\$/\mathrm{kWh}_t$) and feasibility of scaling-up synthesis to large volumes. Such topics of engineering significance are often neglected in fundamental research but are key to the commercial success of nanofluids, if this technology were to penetrate conventional and novel engineering applications. The engineering economics of nanosynthesis, scale-up, and cost will therefore be explored briefly in this review.

6.2 Nanofluid Synthesis Protocols

Nanofluids are typically synthesized by either two-step method or one-step method.

The two-step method, as the name suggests, consists of two separate processes for synthesizing nanoparticles (or commercially procured) and dispersing the procured nanoparticles into base fluid. Such methods are being extensively used in nanofluid research due to its simplicity. Proper choice of synthesis protocol can enable better stability as well as control over precision and size of nanoparticles in the suspension. Nanomaterials used in this method are usually procured commercially, typically in the form of dry powders. With advances in synthesis techniques for nanoparticles, large-scale production of nanoparticles with good precision in size and shape has been achieved by commercial vendors (e.g., using combustion synthesis techniques). The techniques used for making nanoparticles include mechanical methods (milling, grinding, etc.), physical methods (physical vapor deposition, inert gas condensation, etc.), and chemical synthesis (sol-gel process, solution combustion, electrolysis, combustion synthesis, etc.). Depending on the requirements of the chosen applications, different synthesis protocols can be selected to deliver the specification for nanomaterials with the desired constraints for size and shape. This review will be limited to discussion of dispersion protocols rather than the synthesis of nanoparticles in the form of dry powders. Excellent reviews are available in the literature on the topic of the nanoparticle synthesis (e.g., by C. N. R. Rao [2]). These synthesis protocols for nanoparticles in the form of dry powders are categorized into: (a) top-down, and (b) bottom-up techniques. The reader interested in this topic can consult this reference (and similar reviews available in the literature).

The primary bottleneck of the two-step method is that appropriate dispersing technique is needed to ensure the stability of the nanoparticle suspensions in the solvent/fluid phase. Due to the high surface energy nanoparticles inherently form unstable suspensions in liquids and have a strong tendency to agglomerate (which is primarily caused by Brownian motion mediated collision of nanoparticles in the suspension). Various strategies are used for mitigating mutual collision of nanoparticles in the suspension and preventing (or minimizing) the tendency for agglomeration. Typical methods used for preventing agglomeration of nanoparticles include ultrasonic agitation, additives to control pH (e.g., buffer solutions), and functionalization of the nanoparticle with surfactants or chemical groups (such as amines or silanes) to promote better dissolution in the solvent phase by hydrogen bonding and/or ionic interactions. Ultrasonic agitation often provides limited payback since the nanoparticle suspensions typically settle and precipitate from the solution under gravity. The remaining approaches are intrusive methods since they often cause significant alteration of the chemical composition of the solvent phase. The details of nanofluid stabilization will be discussed in the later section. However, the two-step method often fails to deliver long-term stability of the synthesized nanofluids.

The one-step method relies on the generation of nanoparticles in-situ in the solvent phase from precursors. In other words, the synthesis and dispersion of nanoparticles happen simultaneously in the solvent phase. As a result, the propensity of agglomeration of the nanoparticles generated in-situ is minimized. The one-step method can be implemented by either a physical technique (e.g., direct evaporation and condensation) or chemical technique (e.g., chemical decomposition). However, it is more difficult to control the morphology of the particles precisely as small variations in the designed synthesis conditions (temperature, time, feeding rate, etc.) can drastically alter the properties of the synthesized nanofluids due to variation in nanoparticle size distribution and stability. Thus, it is very important to understand, model, and optimize the synthesis conditions to enable better control over the transport mechanisms (e.g., homogeneous or heterogeneous nucleation of the nanoparticles from the precursors as well as growth and subsequent agglomeration of the nanoparticles generated in-situ). It should be noted that if the generation of the nanoparticle is a distinctly separate process from the dispersion step, such methods are categorized as a two-step process.

6.2.1 Two-Step Method

For most nanofluid synthesized via two-step method, commercial nano-powders supplied by manufacturers were used directly. The preparation process itself is rather straightforward: the nanoparticles are first dispersed in the base fluid and then stabilized by different approaches. However, depending on the material of the nanoparticle and base fluid, the dispersion process could be either “spontaneous” or “non-spontaneous”. In the former case, the nanoparticles would readily spread out in the base fluid and remain in the stable suspension state, while in the latter case, the nanoparticles tend to stay together unless external forces are applied. Such variation gives rise to the difference in the nanofluid preparation procedure.

In general, the nanofluids tend to be more stable if there is a strong affinity between the nanoparticles and liquid molecules and a strong repulsive force between nanoparticles. A good example for illustrating particle–solvent interaction would be the dispersion of TiO_2 in water. Due to the proximity of acidic and basic sites on the surface of different TiO_2 crystals (with/without defects), water molecules are found to get absorbed and dissociated on TiO_2 surfaces, with hydroxyl groups binding to surface Ti atom and H atoms binding with the bridging O atom [3–6]. Such feature allows the TiO_2 nanoparticles to form strong bonding with the water solvent, which reduces their possibility of coagulation in the suspension and enhances the stability of the nanofluid consequently. The dispersion of CNTs in water, on the contrary, goes to another extreme. Carbon by nature is almost purely aromatic and non-polar. They possess strong van der Waal forces between each other and high level of hydrophobicity. Thus, CNTs have a strong tendency to form agglomerates with the neighboring groups in common organic and aqueous media unless coated with some stabilizing agent/function group [7, 8].

Strong repulsive forces could also help keeping nanoparticle apart from each other, thereby increasing their stability in suspensions. Such forces could come from the electric double layer (charged stabilization) or absorbed polymeric molecules (steric stabilization) on the particle surface. Adjusting pH is a typical method for enhancing nanofluid, as the higher concentration of H^+/OH^- in the system increases surface charge density, and thus brings higher electrostatic potentials between particles [9]. Adding appropriate ionic compound could induce similar sterilization effect, as the ions transfer their charges to the nanoparticle surface and hence increase electrostatic stabilization [10].

The intermolecular potentials have a direct impact on the collision frequency of nanoparticles in the liquid environment. Apart from this, the stability of nanofluid is also dependent on the probability of merging upon collision, which is closely linked to the specific surface energy of the nanoparticles. From chemical thermodynamic point of view, a high specific surface energy is representative of an unstable state, in which the particles will try to minimize its free energy by forming large aggregates and reduce surface areas. For example, conducting (metal) nanoparticles possess very high specific energy of metal surfaces (1000~2000 mJ/m^2) in comparison with other organic and inorganic materials (~20 mJ/m^2 for teflon and 462 mJ/m^2 for silica) [11]. This results in high instability of "original" metallic nanofluids such that the freshly dispersed nanoparticles would agglomerate and precipitate readily in short time [12].

The point here is that the preparation of nanofluid via two-step method is more than simply mixing the particles with the fluid. Considerable efforts should be focused on how to obtain "stable" nanofluid with additional procedures before, during, and after the mixing process. Such practices are determined by the materials used in nanofluid preparation as well as the experimental conditions. Here, we will briefly summarize the two-step process used for preparing different nanofluids. More details on the different approaches for enhancing nanofluid stability will be discussed below. A general compilation of two-step nanofluid preparation can be found in the appendix for readers' reference.

Typical nanoparticles used in nanofluids are metallic (Cu, Ag, Au, Al, Fe), ceramic (CuO, Al_2O_3, Fe_3O_4, SiO_2, TiO_2, ZiO_2, AlN, SiN), or carbon-based (single/multi-walled CNT, graphene, C_{60}). The typical base fluid used include water/water-soluble molecular liquid (water, ethylene glycol, ethanol), oil (PAO, silicon oil, engine lubrication oil), and ionic liquid ([Bmim][PF_6], [HMIM]BF_4, molten salt). The table below summarizes the stability level of the corresponding nanofluids without any stabilizing agent/surface functionalization. It represents the inherent dispersibility of the nanoparticles in base fluids (Table 6.1).

Table 6.1 Stability of nanoparticles in different fluids

Without treatment	Water/water-soluble molecular liquid	Oil	Ionic liquid
Metallic	Poor	Moderate	Good
Ceramic	Moderate	Good	Very good
Carbon	Very poor	Poor	Moderate

6.2.2 One-Step Method

Although two-step method has been used widely in the nanofluid research community, the issue of agglomeration in the mixed nanofluid has promoted the exploration of the one-step method in which the synthesis and dispersion of nanoparticles are performed simultaneously. Depending on the nature of the synthesis approach, they could be classified into either physical or chemical method.

6.2.2.1 Physical Methods

The advantage of physical synthesis methods in comparison with chemical processes is the absence of solvent contamination in the process of nanoparticle generation. Evaporation-condensation is a typical physical approach for preparing nanofluids in one step, but other methods are also available.

Physical Vapor Deposition

Physical vapor deposition is a broad category of methods used for nanomaterial synthesis, in which nanoparticles were formed by direct condensation of the target metal vapor in contact with a flowing liquid. Different particle concentration and diameter can be achieved by controlling the vapor release and liquid flow rates. Such methods originated from the gas evaporation method used for preparing fine metal particles in the inert gas environment [13], and has been improved and adapted for producing monodispersed nanoparticles in the liquid environment. Akoh [14] synthesized ferromagnetic metal oxide (Fe_3O_4, CoO, and NiO)-based nanofluid by the so-called VEROS (Vacuum Evaporation onto a Running Oil Substrate) method in which particles were generated in oil with an average diameter of 2.5 nm. Wagner [15] prepared silver and iron nanofluids in oil using a similar approach involving magnetron sputtering. Eastman [16] prepared Cu/ethylene glycol nanofluid by evaporating the source metal into cooled liquid using resistive heating (10 nm Cu nanoparticles were produced in EG with 0.5% volume concentration and little agglomeration was observed).

The nanofluid synthesis via conventional PVD chamber requires low vapor pressure base fluid and high power for metal vaporization. Localized high temperature/heat flux technique can be used for achieving more efficient and convenient nanofluid synthesis. Exploding wire method (also known as pulsed wire explosion, pulsed-wire evaporation method) is one common technique used for creating metal vapor with localized high energy input [17]. Lee [18] used pulsed-wire evaporation technique for preparing ethylene glycol-ZnO nanofluid. In their experiment, a pulsed high-voltage DC power was used to induce a non-equilibrium overheating in a thin Zn wire. The pure metal evaporated within microseconds and condensed into small-size particles (<100 nm) after coming into direct contact with the EG. Park

[19] synthesized three different metallic nanofluids including Ag, Cu, and Al nanoparticles in three kinds of fluids: water, ethanol, and ethylene glycol using the electric explosion method. The author reported that higher energy leads to the decrease in the size of metallic nanoparticles. Similar methods were also used for preparing copper [20–22], silver [22–25], iron [22], gold [26], alumina [27], and titania [28] nanofluids.

Nanofluid synthesis with the aid of plasma is also a promising approach due to multiple advantages of this method—including the simplicity of the experimental design. The plasma could be generated either in the air or in the fluid. Chang [29] fabricated Al_2O_3 nanofluid by a modified plasma arc system. In the system, bulk metal was vaporized by high-temperature plasma arc and cooled by pre-condensed deionized water. The rapid cooling process prevented the growth of particle size which produced stable nanofluid with fine particles (25–75 nm). Teng [30] prepared organic nanofluid using a similar system, in which carbon was vaporized in a plasma chamber and cooled by deionized water to form fine carbon nanoparticles (244–284 nm). It should be noted that if pure metallic nanofluids are desired, the plasma should be generated in an inert gas environment to avoid the metal vapor from being oxidized.

Nanofluids can also be prepared directly by solution plasma in which the target material gets vaporized and condensed instantly. For submerged arc plasma methods, a selected metal is heated and vaporized by arc sparking between two electrodes immersed in dielectric liquids. The metallic aerosol then immediately condenses into nanoparticles under the cooling effect of the flowing liquid. Tsung [31] first used the arc-submersed system for synthesizing copper nanoparticle suspensions in de-ionized water (DIW). Cu nanoparticles with either coarse or fine bamboo leave structures (<200 nm) were generated by changing the environmental pressure during synthesis. The technique was adopted and improved for preparing stable TiO_2 [32], CuO/Cu_2O [33], silver [34], and nickel [35] nanoparticle suspension by the same research group, in which nanoparticles with different morphologies were manufactured by changing electric current, arc pulse-duration/off time and dielectric liquids. Saito and Akiyama [36] have made a thorough compilation of available nanomaterial synthesis techniques using solution plasma, where the source materials, reaction media, and electrode configurations are discussed in detail.

Laser ablation in the liquid is another simple and effective way of vaporizing metal solids and synthesizing nanofluids in one step. The method works by focusing a high-power laser at the submerged solid surface for an appropriate time until the solid melts and vaporizes above ablation threshold. Meanwhile, a thin liquid layer near the solid surface will also vaporize with the metal. The expansion of liquid and conversion to vapor fractures the metal into nano-sized drops, which are later supercooled by the surrounding liquid and transformed into nanoparticles [37]. Phuoc [38] synthesized Ag-deionized water nanofluid using multi-beam laser ablation (the synthesized nanoparticles were observed to be stable for several months). It was found that both laser intensities and multi-beam ablation can increase the ablation rate and promote the reduction of the nanoparticle size. Kim [39] prepared bare Au-water nanofluid using single-pulsed laser beam ($\lambda = 532$ nm). The average size of the nanoparticles ranged from 7.1 to 12.1 nm and the nanofluid samples were observed to be stable for 3 months after synthesis. The volume concentration of the synthesized nanofluids was 0.018%. The one-step laser ablation technique has also been used to synthesize a variety of nanofluids with different nanoparticles including: Cu [40], Al [41], Sn [42], Si/SiC [43], CuO [44], Al_2O_3 [45], and carbon particles [46, 47]. Compared to other methods, laser ablation in the liquid is a rather simple and "green" (environmentally friendly) technique for synthesizing nanofluids in water or other organic liquids under ambient conditions. More details on the fundamental mechanism and fabrication process of nanomaterials via laser ablation in liquid were reported by Zeng [48].

Wet Mechano-Chemical Techniques

The top-down approach for synthesizing nanoparticles via purely mechanical actuation (i.e., crushing, milling, and grinding) is an economical, simple, and environmentally benign alternative for synthesizing various nanofluids. With the extensive development and use of high-energy ball milling (HEBM), synthesis of ultrafine nanoparticles has been proven to work for a number of materials [49–51]. Inkyo [52] prepared a well-dispersed suspension of TiO_2 nanoparticles in methyl methacrylate (MMA) with 5% mass fraction using beads milling and centrifugal bead separation. Particle size distribution between 10 and 50 nm was achieved using 660 min milling time, and the nanoparticles remained in stable suspension with no sedimentation after 24 h. Harjanto [53] prepared TiO_2-water nanofluids through the one-step process in which titania nanoparticles were milled together with distilled water in a vial placed in a planetary ball mill. The concentrated nanofluid solution synthesized in this process was diluted into different concentrations and stabilized using ultrasonic stirrer and oleic acid served as a surfactant. The average nanoparticle size was in the range of 24.1–27.2 nm and good stability was confirmed from the measured values of absolute zeta potential. Nine [54] prepared well-dispersed Cu/Cu_2O-water nanofluid using low energy ball milling in aqueous solution by varying the ball size and milling period. Samples that have been ground for 30 min by 1 mm balls and for 60 min by 3 mm balls were reported as stable colloids after performing sedimentation tests for over 7 days. Almasy [55] prepared ferrofluids with particle size of 10–15 nm using vibrating ball mill with industrial magnetite powder. It was observed that the wet-milled magnetite suspension had a higher saturation magnetization than that obtained in the relatively rapid co-precipitation synthesis. Wet mechanochemical methods may be preferred in certain practical situations for preparing nanofluids due to their simplicity, but scaling-up these synthesis techniques for large-scale manufacturing is still challenging.

6.2.2.2 Chemical Methods

One-step synthesis of nanofluids via chemical methods involves the generation and dispersion of target nanoparticles using a single-step or a series of chemical reactions (i.e., reduction, decomposition, etc.) from certain precursors. The process is usually performed in liquid environments and is known as wet chemistry method. Compared to the physical methods, wet chemistry routines are generally cheaper, require minimal instrumentation and are easier to implement, especially for scaling-up these synthesis techniques for large-scale manufacturing as well as for mass production. Also, very precise control over monodispersed nanostructures is achievable by proper choice of the chemistry of the synthesis protocols.

Chemical Reduction Method

Chemical reduction method has been used for nanofluid preparation by a number of researchers. Liu [56] prepared Cu-water nanofluid using copper acetate as precursor and hydrazine (N_2H_4) as reducing agent. The nano-suspension with monodisperse Cu particles (50–100 nm) was formed by slowly adding a predetermined quantity of hydrazine solution into copper acetate solution by constant stirring. Garg [57] synthesized copper nanofluids in ethylene glycol using a similar method except for the addition of sodium hydroxide as an additional reactant. The mean nanoparticle size was measured to be 200 nm. Kumar [58] prepared stable non-agglomerated copper nanofluids by reducing copper sulfate pentahydrate with sodium hypophosphite as reducing agent in ethylene glycol as base fluid by means of conventional heating. The process was fast and cost-competitive. Shenoy [59] employed a different one-step reduction routine for preparing copper nanofluids, in which copper nitrate was reduced by glucose in the presence of sodium lauryl sulfate. The nanofluid was found to be stable for a minimum of 3 weeks, and the method was reported to be reliable, simple, and cost-effective.

Other types of nanofluids have also been synthesized using chemical reduction methods. Tsai [60] prepared aqueous gold nanofluid by the reduction of aqueous hydrogen tetrachloroaurate ($HAuCl_4$) with trisodium citrate and tannic acid. Au nanoparticles with different sizes were obtained by changing the amounts of tetrachloroaurate, trisodium citrate, and tannic acid. Xun [61] prepared stable silver nanofluid in kerosene by first extracting silver nitrate in nonpolar solvent by thio-substituted phosphinic acid extractant Cyanex 302, and then reducing Ag^+ solid. Salehi [62] prepared silver nanofluids using a different approach, in which silver nitrate was reduced by sodium borohydride and hydrazine using polyvinylpyrrolidone (PVP) as the surfactant. It should be noted that the chemical reduction method only works for a very limited number of metallic particles which are chemically inert.

Precipitation Method (Ion Exchange)

Nanoparticles can be synthesized in various liquids by interactions between different ions at a controlled rate. Cao [63] prepared 5 nm ZnO nanorods in ethanol by adding sodium hydroxide into zinc acetate dehydrate ($Zn(Ac)_2 \cdot 2H_2O$) solution at room temperature. It was found that the size and shape of nanorods with different size and shapes can be tuned via simply altering NaOH concentration and reaction time. Darezereshki [64] prepared maghemite (γ-Fe_2O_3) nanoparticles by mixing the $FeCl_3$/$FeCl_2 \cdot 4H_2O$ solution and the$NH_3 \cdot H_2O$ solution with vigorous stirring for 2 h. The average particle size was observed to be 45 nm. Manimaran [65] prepared copper oxide nanofluids by mixing copper chloride with sodium hydroxide in deionized water along with heating and magnetic stirring. The precipitates were observed to have an average size of 20 nm with very little agglomeration. Chakraborty [66] synthesized Cu-Al layered double hydroxide nanofluid via the one-step method. The nanoscale precipitation was formed by the dropwise addition of NaOH solution into the aqueous mixture solution of $Cu(NO_3)_2 \cdot 3H_2O$, $Al(NO_3)_3 \cdot 9H_2O$, and $NaNO_3$. The particles were observed to form clusters with size ranging from 86 to 126 nm in the suspension, and the nanofluid was found to be stable with high zeta potential value. Nanoparticles were also synthesized by the precipitation method first and re-dispersed in the fluid later after additional treatment (i.e., centrifugation, washing, calcination) [67–70]. Such methods should not be categorized as the one-step method as the synthesis and dispersion processes were not conducted simultaneously.

Sol-Gel Method (Hydrolysis)

Sol-gel process is a widely used technique for synthesizing nanoparticles with different size and scale of gel networks. The reaction mechanism involves two stages: (1) hydrolysis reaction of the precursor in which the functional binders of the precursors are substituted with the hydroxyl group; and (2) polycondensation reaction in which the hydroxyl group of monomers gets connected and forms continuous network [71]. Most nanofluid literature involving sol-gel process have adopted a two-step method for preparing the nanofluid samples, in which the nanoparticles were first generated from precursors, separated out from the reacting liquid, calcinated, and re-dispersed into the targeting base fluid [72–78]. However, there are also few cases where the nanofluid is prepared directly via the sol-gel method. Kim et al. [79] prepared stable silica nanofluid in water using TEOS as precursor, ethanol as solvent, DI water for hydrolysis reaction, and ammonium hydroxide as base catalyst. The particle size was approximately 30 nm, 70 nm, and 120 nm by controlling the ammonium hydroxide concentrations at 0.28 mol, 0.42 mol, and 0.56 mol, respectively. Jing [80] prepared highly dispersed silica-water nanofluid samples using a similar one-step sol gel process. Nanofluids with particle sizes of 5, 10, 25, and 50 nm were fabricated by first bending H_2O, NH_3 and alcohol, and then adding

TEOS with different quantities. The main issue with preparing nanofluid by sol-gel in one step is the contamination from excess reacting chemicals.

Emulsion-Polymerization

Emulsions are dispersed systems with two immiscible liquids (i.e., oil and water) in which liquid droplets are dispersed in a liquid medium. When micro/nano-emulsion materials are mixed, the reactant exchange from the dissolved molecules in each liquid could potentially promote precipitation reactions in the nanodroplets, which is then followed by nucleation, growth, and polymerization of the nanoparticles [81]. Han [82] developed a one-step, nanoemulsification method to synthesize the indium/polyalphaolefin (PAO) nanofluid. In the test, an indium pellet was first added to the PAO oil in a reaction vessel heated to 20 °C above the indium melting temperature. A PAO aminoester dispersant was injected into the reaction vessel, which also acts as a stabilizer to prevent nanoparticle coagulation. The emulsion was then exposed to high-intensity ultrasound radiation for more than 2 h until a stable nanofluid was formed. Kim [83] prepared biphasic nano-colloids of poly(dimethyl siloxane) (PDMS) and an organic copolymer (methyl acrylate co-methyl methacrylate co-vinyl acetate) in aqueous solution via emulsification and polymerization route. The particles were observed to have 170 nm size with a spatial resolution of 8 nm achieved in the STEM images. Pattekari [84] prepared stable nano-encapsulation of poorly soluble anticancer drug in water using a sonication assisted layer-by-layer polyelectrolyte coating (SLbL). In the experiment, polyanion solutions were added into the drug powder-contained DI water. The process involves simultaneous breaking down of the drug powder by ultrasonication and formation of polycation/polyanion shell by polymerization. The average size of the encapsulated particles was in the 100–200 nm range. A similar method was adopted by Lvov [85] for producing aqueous nanocolloids of encapsulated drug particles with 150–200 nm diameter. It should be noted that nanoscale synthetic polymer in solution has been widely used in biomedical applications. The more frequently used terminology in biotechnology literature is "nanocolloid", but the concept is essentially same as nanofluids.

Microwave-Assisted Reaction

The use of microwave irradiation has been adopted in various studies for promoting nanoparticle formation in liquids via chemical reactions. Such methods were found to be fast and efficient for preparing nanofluids in one-step synthesis protocols compared to other chemical routes. Zhu [86] prepared copper nanofluids by reducing $CuSO_4 \cdot 5H_2O$ with $NaH_2PO_2 \cdot H_2O$ in ethylene glycol under microwave irradiation. Most of the Cu particles are about 10 nm in diameter, and the nanofluid was found to be stable for more than 2 weeks in the quiescent state at 120 °C. Nikkam [87] fabricated Cu nanofluids in diethylene glycol (DEG) base liquid, by heating up $Cu(Ac)_2 \cdot H_2O$–DEG mixture in microwave oven with PVP as a stabilizer. The average nanoparticle size produced was 75 ± 25 nm. Singh [88] prepared stable silver nanofluid in ethanol by reduction of $AgNO_3$ with polyvinylpyrrolidone (PVP), used as the stabilizing agent, using microwave radiation. Ag nanoparticles with size ranging from 30 to 60 nm were produced with different salt-to-PVP ratio and microwave irradiation duration. Habibzadeh [89] prepared SnO_2 nanofluid in water by the microwave-induced chloride solution combustion synthesis (CSCS) method, in which $SnCl_4$, sorbitol, and ammonium nitrate were heated up to combustion temperature by microwave irradiation. The average particle size was 69 and 153 nm for two different samples. Jalal [90] synthesized zinc oxide nanoparticles in ionic liquid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide, [bmim][NTf_2], by microwave decomposition of zinc acetate precursor.

Microwave irradiation can provide a rapid synthesis technique by uniform heating of reagents and solvents which helps to accelerate the chemical reaction of the metal precursors as well as the nucleation of nanoparticles in the solution. Such features result in monodispersed nanostructures which is beneficial for nanofluid synthesis.

Other Methods

Researchers have also considered various other approaches for generating nanoparticles in base fluids. Teng [91] developed an oxygen-acetylene flame synthesis system to fabricate nanocarbon-based nanofluids (NCBNF) through a one-step method. In the system, the O_2-C_2H_2 flame was served as a carbon source, and the generated smoke was cooled and condensed by water mist to form NCBNF. Kim [92] developed an one-step electrochemical method for producing water-based stable carbon nano colloid (CNC) without adding any surfactants at the room temperature. Carbon nanoparticles were formed by applying electric power to the graphite anode and stainless steel cathode immersed in a DIW bath. The samples were observed to be stable for 30 days and the average size of the suspended nanoparticles was measured to be ~15 nm. Phase transfer method has been developed for preparing homogeneous and stable graphene oxide colloids. Graphene oxide nanosheets (GONs) were successfully transferred from water to n-octane after modification by oleylamine. Kang [93] prepared high-quality carbon nanotube in water by dipping red-hot (> 800 °C) graphite rods into cool water repeatedly. The multiwall nanotubes synthesized in-situ were found to have an inner diameter between 5 and 10 nm and outer diameter between 30 and 50 nm.

6.3 Nanofluid Stabilization

Stabilization is the most important issue in nanofluid research and applications, as the properties of nanofluids could be drastically affected by the clustering and aggregation of nanoparticles. Clustering of nanoparticles in fluid is a natural and spontaneous process due to the strong Brownian motion of liquid molecules which promote collision between nanoparticles, while the high surface energy of nanoparticles promotes adhesion after collisions [94]. In these clusters, the nanoparticles could be held together by either weak physical bonds (intermolecular forces) which are readily broken apart by external forces, or tight chemical bonds (which are difficult to separate). In the previous case, the process is usually reversible as the size of cluster can be reduced easily by means of ultrasonication or stirring. In the latter case, the clustering process is irreversible as very strong forces (i.e., high energy ball milling) are needed to break down the agglomeration [95]. In many cases, nanoparticle precipitates are "weak" agglomerates which can be easily re-dispersed into fine nano-suspensions. In other cases, stable agglomerated particle clusters could still be dispersed uniformly in the suspensions for long times without sedimentation. Hence, the concept of "stable" nanofluid should be clarified into different applications and scenarios.

6.3.1 Characterization of Stability

Although there is no standardized protocol for quantitatively evaluating the level of stability of particle dispersion in nanofluid/noncolloid, three methods have been generally used by different researchers for characterizing and analyzing the stability of nanofluids.

6.3.1.1 Sedimentation and Centrifugation Methods

Sedimentation method is the simplest and most straightforward method for characterization of nanofluid stability. Certain quantity of nanofluids is dispensed into a specific container and the process of nanoparticle sedimentation in the suspension is observed over time. In most studies, a time frame will be reported as an indicator for the stable duration for the sample nanofluid, during which no or little visual sedimentation of particles can be observed visually [57, 96–111]. Many of these studies have also recorded the particle precipitation process using cameras, while few other studies have tried to measure the particle sedimentation quantitatively by recording the drop in solid–liquid interface height [112], thickness of particle sedimentation [113], nanoparticle densities at different height in the nanofluid [110], etc. One significant drawback of the sedimentation method is that it could be extremely time-consuming as some nanofluids were found to be stable over 12 months [114]. In order to evaluate the nanofluid stability faster, centrifugation methods have also been used by a variety of studies, in which visual investigation of sedimentation of nanofluids was performed using a dispersion analyzer centrifuge. Singh [88] confirmed the stability of silver nanofluid by centrifuging the nanofluid sample at 3000 rpm for 10 h. Mehrali [115] prepared graphene nanoplatelets (nanofluids) in distilled water with different mass concentrations. The author observed little sedimentation on the bottom of test tubes after the samples were centrifuged at 6000 rpm for 5–20 min. Fang [116] prepared deep eutectic solvent-based graphene nanofluids and confirmed the stability by centrifugal process (5000–20,000 rpm) for 30 min.

6.3.1.2 Zeta Potential (Electro Kinetic Potential)

Zeta potential is the electrostatic potential between bulk fluid and particle surface induced by the particle surface charge. This indicates the interaction energy between particles, and is in many cases responsible for the stability of particles toward coagulation [117]. Generally speaking, high absolute value of zeta potential means stronger repulsive force between nanoparticles, and hence indicates better stability of nanofluids. Typically, a colloid is considered unstable, moderately stable, stable and highly stable with zeta potential values less than 30 mV, between 30 and 40 mV, between 40 and 60 mV, and greater than 60 mV, respectively [118]. Measurement of zeta potential is a well-developed technique with standardized protocols [119]. The process itself is fast and easy through a variety of commercially available equipment. Hence, this is widely used for characterizing the stability of nanofluids under different conditions [53, 66, 97, 104, 106, 120–122].

6.3.1.3 Spectral Absorbency Analysis

In typical spectral absorbency analysis, the intensity of radiation absorption passing through a target sample is measured for different frequencies. It is possible to characterize the particle size distribution of nanoparticles in nanofluid using absorbance spectrum, since the optical properties of small nanoparticles depend on their morphology (i.e., size and shape). UV–Visible spectroscopy has been adapted for use as a simple and reliable method for monitoring the stability of various nanoparticle solutions including gold [123], copper [124], Al_2O_3 [124–126], ZnO [127], CuO [128], SiO_2 [128], TiO_2 [89, 125, 129], CNT [130], etc. The analytical prediction of the particle size from the spectrum could be achieved using the well-known theory of Mie by Kreibig and Genzel [131]. In general, as the particles become less stable (agglomerate or precipitate), the original extinction peak will decrease in intensity due to the depletion of the fine stable nanoparticles, and often the peak will broaden or a secondary peak will form at longer wavelengths due to the formation of aggregates.

6.3.1.4 Electron Microscopy and Dynamic Light Scattering

One straightforward approach for monitoring the nanofluid stability is to measure the particle size at different time intervals. Scanning/transmitting electron microscope (SEM/TEM) can capture fine image of nanoparticles with resolution down to nanometer scale. The distribution of particle size and evolution of particle coagulation can be directly visualized by observing the particle images. Dynamic light scattering is another commonly used technique for determining particle size in suspension/solution. The process is fast and easy and does not require separation of nanoparticle from the solvent.

6.3.2 Approaches for Enhancing Stability

6.3.2.1 Mechanical Mixing

Mechanical mixing has been widely used in nanofluid preparation for attaining better stability. It is a fast and efficient way for breaking down agglomerated clusters and keeping individual particles separated from each other.

Ultrasonication

Ultrasonication is one of the most commonly used mechanical mixing methods for improving the dispersion stability of nanofluids, in which the longitude sound wave travels through the nanofluid suspension and induces strong oscillations of molecules in the system. Such agitated motion promotes distortion of nano-agglomerates which breaks them into finer particles. Depending on the type of ultrasonicator used, the sonication wave can be applied by either direct or indirect means. In the direct sonication case, a sonication probe is immersed directly into the suspension and the sonication energy is released into the liquid without physical barriers. In the indirect sonication case, the colloidal mixture is usually contained in a vessel which is partially immersed in a sonication bath. The sonication wave was generated on the surface of the bath or chamber, which then travelled through the bath liquid and passed through the container wall before it finally reached the suspension sample. The detailed procedure for performing ultrasonic dispersion of nanofluids was discussed in an NIST protocol by Taurozzi and Hackley [132], in which they have recommended direct sonication over indirect sonication due to the higher effective energy output. However, there is not enough experimental data in support of such claims, as considerable number of reports have used sonication bath for preparing nanofluids which showed good stability over time. Nevertheless, since appreciable amount of sonication energy was absorbed by the suspension container, it generally requires more time and power for bath-sonicator (indirect) for achieving good dispersion compared to the probe-sonicator (direct). As significant amount of heat is released in the ultrasonication process, a shorter sonication period could potentially lower the risk of nanofluid degradation due to overheating, unless a cooling system is incorporated in the ultrasonicator. Generally speaking, the maximum duration of probe sonication is in a time scale of few minutes, while the maximum duration of bath sonication can be extended to more than 24 h.

As stated in the NIST protocol [132], sonication is a highly system-specific dispersion procedure, and suggested that the optimum parameter for sonication power and time varies from sample to sample. The determination of the optimum sonication parameters is a trial-and-error process, in which the researcher should start by referring to literature studies of similar particle–liquid combinations. However, it should be kept in mind that even with the same type of material and particle concentration, the optimum ultrasonication time could be different due to the difference in experimental conditions.

High-Pressure Homogenizer (HPH)

HPH is a powerful and effective method to produce homogenous particle dispersion in liquid, by forcing the nanofluid flowing through a narrow valve under high-pressure conditions. Typical processing pressure of HPH ranges between 20 and 100 MPa, in which the high shear stress ruptures large agglomerates into small and fine particles. The effect of pressure on nanofluid stability has not been studied thoroughly, but it has been shown in few studies that nanofluid prepared via HPH exhibits smaller particle size and hence better stability compared to ultrasonication and other mechanical approach [133, 134]. Hwang et al. [133] examined the TEM images of carbon black (CB) nanoparticles in water-based nanofluid stabilized using stirrer, ultrasonic bath, ultrasonic disruptor, and HPH. It was found that only HPH method can provide sufficient energy to break the particle clusters. Fedele et al. [134] examined average particle size of three different nanofluid samples ($CuO/TiO_2/SWCNH$ - H_2O) dispersed via ball milling, ultrasonication and HPH at 4 and 15 days after synthesis. It was observed that nanofluid stabilized with HPH has the lowest level of agglomeration. However, it is worth noting that very few studies have used HPH for synthesizing nanofluids in which the stability enhancement was only verified in small time scales (less than a month). It is not clear if PHP is really more effective in preventing nanoparticle agglomeration in longer term compared to that of ultrasonication approaches.

6.3.2.2 Dispersant

The addition of dispersants (also referred to as surfactants) is an easy and economic approach for enhancing the stability of nanofluids. The dispersing agents are usually amphiphilic organic molecules with both hydrophobic tail and hydrophilic head group. These dispersants will attach to the surface of the nanoparticle due to the mutual affinity, which helps increase the contact at the interface between the solid particle and base fluid. In addition, the tail of the attached dispersant works as a steric barrier which prevents the particles from agglomerating. Such effect is known as steric hindrance and inhibits the coagulation of nanoparticles in the suspensions. The absorbed layer also enhances zeta potential and promotes electrostatic stabilization effect [135].

The selection of suitable dispersant is dependent on the particle and base fluid. It is suggested that water-soluble surfactants should be used if the base fluid is the polar solvent, while oil-soluble surfactants should be used if the base fluid is non-polar [136]. Such characteristics are usually represented by the hydrophilic–lipophilic balance (HLB) value of the surfactant, which describes the balance of the size and strength of the hydrophilic and hydrophobic groups of the surfactant. It has been reported that surfactants with HLB values greater than 10 have the higher affinity in aqueous solutions, while those with HLB values less than 10 are more oil soluble [137]. One example of oil soluble surfactant is oleic acid, which has extremely low HLB value of one and has been extensively used in the preparation of non-polar nanofluids (i.e., transformer oil, silicone oil, kerosene, etc.). The typical example of water-soluble surfactants is sodium dodecyl sulfate (sodium lauryl sulfate, SDS) with a high HLB value of 40. It has been used in the preparation of various kinds of water-based nanofluids.

Still, it should be noted that the choice of the surfactant does not necessarily have to be consistent with the HLB-based principle. Parametthanuwat et al. [138] prepared aqueous silver nanofluid using oleic acid as dispersing surfactant, which showed effective enhancement of the nanofluid dispersion. Also, most surfactants are organic chemicals which easily degrade at elevated temperatures. Hence, the use of surfactants should be performed in accordance with the actual experimental requirements.

6.3.2.3 pH Control

Adjusting pH value can significantly improve the stability of the nanofluids by changing the zeta potential of the system. By moving away from the isoelectric point (IEP), the surface charge of nanoparticles increases due to the more frequent attachment of surface hydroxyl group. The highly ionic charged state effectively keeps particles apart and hinders agglomeration due to the mutual electrostatic repulsive forces. In practice, such adjustment is usually performed with careful addition of acidic or alkaline chemicals (i.e., HCl or NaOH). For each different nanofluid mixture, there exists an optimum pH condition, at which the absolute value of zeta potential is maximized to ensure that the most stable conditions for the nanofluids are attained. For example, the maximum zeta potential for Cu-H_2O, Al_2O_3-H_2O, and SiC-H_2O nanofluid systems is attained at pH = 8.5~9 [139, 140], pH = 8 [9], and pH = 10 [141], respectively. Such values are dependent on the nanoparticle-fluid system and should be obtained experimentally from parametric studies.

6.3.2.4 Surface Modification

Nanoparticle surfaces can be modified to include different functional groups which enable them to be dispersed in the base fluid. These functional groups could effectively enhance the wettability of the solid–liquid interface, reduce the surface energy of the target nanoparticle and van der Waals forces between particles. Wet chemistry method is the most commonly used approach for performing surface functionalization, in which the target nanoparticle is soaked in the reactant for long enough time at desired temperatures. For example, silica nanoparticles can be grafted with silane groups directly by mixing with trimethoxysilane and stirring vigorously for 48 h at 70 °C [142]. CNTs can be functionalized with carboxyl and hydroxyl groups by submerging in sulfuric/nitric acid solution [143]. These functionalized nanoparticles showed superior dispersion in nanofluids compared to that of their pristine forms. Plasma treatment is another surface functionalization approach, in which the target nanoparticles are functionalized by exposing to a continuous wave of discharged plasma of the desired gas. The technique has been used to coat copper and CNT surfaces with various polar groups which help to enhance their dispersion in water [144, 145].

6.4 Summary

Nanofluids have received tremendous attention in the last 2~3 decades due to their superior properties. They have been reported as promising engineering materials to be used in various applications and industries. However, the exploration of nanofluids has still been mainly limited in laboratory-scale studies. Regardless of the increasing number of research papers and patents relating to nanofluids, the commercialization of nanofluids in the real industrial world has not been successful as the research community anticipated [146]. There are many challenges for application of nanofluids in industries, among which the two most critical issues are the long-term stability of nano-suspensions and the prohibitive cost of nanofluid manufacturing in large quantities. These issues are closely linked with the synthesis process of nanofluids, as it has been shown that the stability of nanofluids is greatly dependent on the preparation procedure. The cost issue of nanofluids is significant in real world application, since most nanoparticles are very expensive comparing to the base fluid. Even at low concentrations (i.e., 1.0%), the material cost of nanofluids could be four or five times that of the base fluid. Consequently, in-situ synthesis of nanofluids from cheap materials/precursors is crucial for practical applications. Despite extensive research on nanofluids, a standard set of procedures for nanofluid synthesis should be developed with proven stability of both morphology and material properties. Due to the complex nature of transport mechanisms in nanofluids, the accomplishment of such goals would require a comprehensive and systematic study for better comprehension and verification of the contradictory research results.

Appendix: Nanofluid Two-Step Synthesis

6.4.1 Metallic Nanoparticle-Based Nanofluids

Metallic nanoparticle-based nanofluids have drawn much interest due to the high thermal conductivity of metals. It is expected that the metallic suspensions can enhance the thermal transport properties of conventional heat transfer fluids which makes them suitable for heat exchanger applications.

Copper Nanofluids

The use of copper nanoparticle in nanofluids is appealing since copper has relatively high resistance to corrosion. Xuan and Li [96] prepared both water-Cu and oil-Cu nanofluids using the two-step method in which copper nanoparticles of about 100 nm diameter are directly mixed in water and mineral oil. For water-based nanofluid, laurate salt at 9% concentration was used as a dispersant which was observed to hold the water-Cu suspension stable for 30 h in a stationary state with minimal amounts of clustering. For oil-based nanofluids, oleic acid at 22% concentration was dispersed in the nanofluid to stabilize the suspension followed by ultrasonication for 10 h. The oil-Cu nanofluid was proved to be stable for 1 week with no sedimentation. Li [97] prepared aqueous copper nanofluid by mixing copper nanoparticle (~25 nm) in purified water at 0.1% concentration (with and without dispersant under different pH conditions). He observed that the addition of CTAB dispersant enhanced the stability of water-Cu suspensions by reducing the diameter of copper nanoparticles from 5560 to 130 nm. The nanofluid sample without dispersants quickly agglomerated, while the sample with dispersant remained stable with no sedimentation after 1 week. The water-Cu nanofluid samples showed maximum zeta potential value at pH = 9.5 which indicates that the suspension exhibits better dispersion in slightly basic environments. Saterlie [147] prepared water-Cu nanofluid by first synthesizing copper nanoparticles (~100 nm) in-situ using chemical reduction method and then re-dispersing them in water. The nanofluid was stabilized by adding SDBS as dispersing agent and ultrasonication for 50 min. He found that by increasing the Cu particle loading from 0.55 to 1.0%, several agglomerates are formed in the nanofluid with nanoparticle size increasing from 120 to 800 nm.

Surfactant-free copper nanofluids were also explored in various studies. Lu [148] prepared surfactant-free water-Cu and ethanol-Cu nanofluids by mixing copper nanoparticles (~20 nm) with the base fluid at 0.2–2% concentration and ultrasonicating for 10 h. The nanofluids were tested in a flat capillary pumped loop and sedimentation of nanoparticles observed on the heated surface. However, the morphology of the working nanofluid was not examined after the test. Kole [98] dispersed copper nanoparticles (~40 nm) in distilled water at 0.5% concentration with ultrasonication for 10 h. The suspensions remained stable for more than 15 days with no significant sedimentation. Garg [57] prepared EG-Cu nanofluids by synthesizing copper nanoparticles using a chemical reduction method, with water as the solvent, and then dispersing them in ethylene glycol using a sonicator. The particle loading is 2.0% and no sedimentation was observed after a few days.

It is difficult to compare these different studies and draw a general conclusion on the effect of dispersing agent and ultrasonication toward the stability of nanofluid samples, since the nanofluid samples are synthesized under different conditions with different material characteristics. The stability of nanofluids is very sensitive to the variation in the size of the nanoparticles, concentration, pH, ultrasonication time, surfactant, etc. It is also important to notice that most studies have only shown stable nanofluids for the limited period (from few hours to 1 week). That suggests preparing stable samples of copper nanofluids via two-step methods to be used in long-term duty-cycles is still a challenging research topic.

Gold and Silver Nanofluids

Gold and silver nanoparticles have also been used in many studies due to their unique optical, electrical, and thermal properties (i.e., high electrical conductivity, stability, and low sintering temperatures). Such properties make them desirable in wide range of applications including diagnostics, antibacterial agents, heat transfer fluids, and optical fluids [149].

Preparation of gold nanofluids via two-step method is rare as most gold nanofluids were synthesized directly in the target base fluid from chemical reduction approach [150–154]. Silver nanofluids can be prepared by mixing manufactured silver nanoparticles in the base fluid. DisKang [155] prepared water/EG-Ag nanofluids at 0.1–0.4% volume fraction by dispersing silver nanoparticles (8–15 nm) into fluids without additives or stabilizers and ultrasonicating for 3 h. The nanofluid was generally stable for 1 day. Oliveira [156] prepared stable water-Ag nanofluid with 0.15% volume loading and 80 nm diameter nanoparticles using high-pressure homogenizer. The stabilization was achieved by placing the mixture in the high-pressure homogenizer and circulating for 30 min at 400–500 bars. The nanofluids were visually verified to be stable for at least 6 months. Hwang [133] prepared silicone oil-Ag nanofluid by dispersing produced silver nanoparticles in the base fluid with the assistance of various physical treatment techniques. With primary particle size of 35 nm and particle weight loading of 0.5%, he found that without any treatment, Ag nanoparticles were highly agglomerated in the pure fluid with an average nanoparticle size of 335 nm. Such values were reduced drastically to 182 nm, 147 nm, 66 nm, and 45 nm, after using stirrer, an ultrasonic bath, an ultrasonic disruptor, and a high-pressure homogenizer, respectively. Warrier [157] prepared water-Ag nanofluid at the concentration of 1 and 2% with nanoparticle size of 20, 30, 50, and 80 nm, respectively. The suspension was stabilized by both polyvinylpyrrolidone (PVP) at a concentration of 0.3% and ultrasonication process (while no settlement was observed for 2 h) after synthesis. Paramethanuwat [158] prepared water-Ag nanofluid at a concentration of 0.5% by repeated magnetic stirring and ultrasonicating after the addition of oleic acid (OA) (at concentration of 0.5, 1, and 1.5%) and potassium oleate surfactant (OAK^+). It was found that the OAK^+ exhibited good adsorption on the silver nanoparticles which helped improve the colloidal stability and non-precipitation period of the silver nanoparticles for up to 48 h.

Gold and silver nanofluids are typically synthesized via one-step method due to their inherent simplicity and competitive costs. It is worth noticing that these nanofluids were shown to be stable over several months—just by physical treatment.

Aluminum Nanofluids

Aluminum nanoparticles are of great interest in a variety of fields due to their high enthalpy of combustion and rapid kinetics. These characteristics make them favorable in fuel engineering field including alloy powder metallurgy parts for automobiles and aircrafts, rocket fuel, igniter, smokes, and tracers [159]. The study of aluminum nanofluid is limited as they are easily oxidized into alumina. Boopathy [160] prepared aluminum nanoparticles (~150 nm) by mechanical milling and dispersing them in distilled water and engine oil with 0.025% volume loading. The nanofluids were stabilized using 1% sodium lauryl (Dodecyl) sulfate as dispersant followed by 10 min of ultrasonication at 20 kHz and for 30 min of magnetic stirring at 1500 rpm. Teipel [161] prepared aluminum nanofluid using paraffin oil and HTPB as the base fluid. The Al particles (~80 nm) were dispersed by stirring for several hours and using ultrasound homogenizer before the suspension was tested for rheological measurements.

Aluminum nanopowders can react with water at high temperature (400–600 °C) to generate hydrogen which enhances fuel combustion [162]. Such feature promotes studies on aluminum nanofluid used in combustion and fuel. Kao [163] prepared aqueous aluminum nanofluid for diesel fuel combustion by producing emulsified nano-aluminum (40–60 nm) liquid using both ultrasonic vibrator and agitator. The work did not discuss the stability of the aluminum suspension. Gan [99] prepared aluminum nanofluid in *n*-decane and ethanol fuels with 80 nm Al nanoparticles at 10% mass loading by stirring the mixture vigorously and ultrasonication in an ice bath for 5 min. He observed that the suspension of *n*-decane/nano-Al remains stable for 10 min while ethanol/nano-Al can last for 24 h without obvious sedimentation. Xiu-tian-feng [164] synthesized stable jet fuel-Al nanofluids with 1.0% mass loading by modifying the surface of aluminum nanoparticles with various chemicals. It was found that oleic acid is the most effective surface modification agent which keeps the suspension stable for more than 2 weeks.

The use of aluminum nanoparticles in fuels and combustion requires high enough concentration for achieving considerable contribution to the energy content. Thus, the stabilization of the nano-mixture suspensions for long enough time is crucial for them to be utilized in liquid fuels.

Iron Nanofluids

Iron nanomaterials are of great interest as iron is among the most useful magnetic materials as well as the most abundant and widely used elements on earth. Doping iron nanoparticles in fluid manifest both fluid and magnetic properties which open new area of applications in electronic device, spacecraft propulsion, material science, biomedical instruments, and so on [165, 166]. Hong and Yang [167] prepared iron nanofluids with ethylene in which the Fe nanocrystalline powder (~10 nm) was first synthesized by chemical vapor condensation process and then re-dispersed in the base fluid with 0.2, 0.3, and 0.4% volume loading using ultrasonication (20 kHz). They observed an increment in thermal conductivity of the nanofluid with increasing sonication time from 10 to 70 min and ascribed it to the improved stability of suspension with prolonged sonication. Sinha [168] prepared EG-iron nanofluid by synthesizing iron nanopowders from chemical reduction method and mixing them in the base fluid under 50 min ultrasonic irradiation in concentrations of 1.0 vol.%. Agglomeration of nanoparticles was observed since the nano-crystallite sizes of the powders were below 20 nm while the average particle size in the fluid was around 500 nm. Xuan and Li [169] prepared magnetic iron nanofluid by directly dispersing Fe nanoparticles (~26 nm) into deionized water with the volume percentage of range from 1.0 to 5.0%. The suspension was stabilized using 1.0–6.0 vol.% sodium dodecylbenzenesulfonate as activator and the nanosamples showed good stability from few hours to 1 week. Gan [170] studied the combustion of iron nanofluid fuels prepared from dispersing iron nanoparticles (25 nm) in *n*-decane/ethanol with 5–20 wt.% concentration by hand mixing and ultrasonication. The nanofluid was stabilized with 0.5 wt.% sorbitan oleate as surfactant and mixture remains suspended for few hours.

Although pure iron exhibits better saturation magnetization, they are highly toxic and very sensitive to oxidation without appropriate surface treatment. In contrast, iron oxide nanoparticles are less sensitive to oxidation and, therefore, can give a better and stable performance [171].

Other Metal Nanofluids

Quite few other metal nanoparticles were also explored for synthesizing energy-efficient nano-suspensions. Naphon [172] prepared titanium nanofluid by mixing 21 nm Ti nanoparticles in de-ionized water and alcohol using an ultrasonic homogenizer. The nanofluid was tested in heat pipe without characterization on the suspension stability. Chopkar [173] prepared Al_2Cu and Ag_2Al dispersed nanofluids using the two-step method, in which Al_2Cu (20–30 nm) and Ag_2Al (30–40 nm) nanoparticles were first produced by mechanical alloying using a high energy planetary ball mill followed by dispersing these particles into ethylene glycol and water with volume fractions from 1.0 to 2.0%. The suspension was homogenized by intensive ultrasonic vibration and magnetic stirring with the addition of 1.0 vol.% oleic acid as surfactant. The sample showed good stability with some tendency of agglomeration during the test. However, metal nanoparticles were found more likely to be oxidized at even low temperature [12] which brings instability in the hydrothermal performance. Besides, metal nanofluids suffer from the issue of quick sedimentation and fouling which makes it challenging to use them in practical applications [174, 175].

Oxide Nanoparticle-Based Nanofluids

Oxide nanomaterials have been intensively used in modern nanotechnology. Their unique physicochemical properties have opened up applications in nanoelectronics, sensors, optics, catalysts, biomedicine, etc. [176–180]. Preparation of nanofluids using oxide nanoparticles via two-step methods is discussed below.

Copper Oxide Nanofluids

Copper oxide nanoparticles are used in diverse applications with a range of useful properties such as high electric/thermal conductivity, electron correlation effect, high atom efficiency, etc. [181, 182]. Choi and Eastman [183] first studied copper oxide nanofluid in water and ethylene glycol, in which they dispersed CuO (~20 nm) nanoparticles produced by gas condensation in the base fluid directly by shaking thoroughly. It was observed that Cu nanoparticles agglomerated into large particles (~100 nm) which could still form the stable suspension in the liquid. Kwak [184] prepared copper oxide nanofluid in ethylene glycol using 10–30 nm Cu nanoparticles at 0.001–1% volume fraction dispersed by ultrasonication. It was found that sonication for 9 h gives the best dispersion and the suspension was stable for 100 days. Namburu [185] prepared copper oxide nanofluid in EG–water mixture (60:40) with a particle size of 29 nm and volume concentration increasing from 1 to 6%. The nanofluid mixture was stirred and agitated thoroughly for 30 min with an ultrasonic agitator for ensuring uniform dispersion. Kulkarni [186] did a similar study by mixing CuO nanoparticles (~29 nm) in deionized water with 5–15% volume fraction. The uniform mixture of nanoparticles in water was attained by thorough stirring and ultrasonicate agitation for half an hour. Li and Peterson [100] prepared water-CuO nanofluid with 29 nm diameter and 2–10% volume fraction. The powder and base fluid were blended by immersing in an ultrasonic bath for 3 h and the suspensions were found to be very stable, with essentially no sedimentation over 7 days. Karthikeyan [101] prepared water/EG-CuO nanofluid using monodispersed CuO particles of 8 nm diameter. The suspension was homogenized by using an ultrasonic horn for 30 min without the addition of surfactant. The study found that the nano-mixture remained stable for more than 3 weeks with particle volume concentration below 1%. Above 1% volume concentration, sedimentation in CuO nanofluid was observed. Rashin [102] prepared copper oxide-coconut oil nanofluid using 20 nm CuO nanoparticles with 0.5–2.5% mass concentration. The nanoparticles were dispersed by only 1 h ultrasonication and the suspension remains stable for 7 days after which the sedimentation starts. Kole [103] prepared stable nanofluid by dispersing 40 nm spherical CuO nanoparticles in gear oil with volume fraction ranging between 0.5 and 2.5%. The mixture was stabilized by mixing with oleic acid, intensive ultrasonication for 4 h and magnetic agitation for 2 h. Although aggregation of CuO nanoparticles was identified with average cluster size ~7 times of the primary diameter, the suspension was stable for more than 30 days without visual sedimentation. Sahooli [120] studied the effect of pH and surfactant concentration on the stability of CuO nanoparticles (4 nm, 1.0 wt.%) in the water-based nanofluid. He proposed that the suspension zeta potential and absorbency were maximized at pH = 8 and 0.095 wt.% PVP, which is the optimum condition for obtaining most stable nanofluid. However, the average particle size measured with PVP surfactant was 63 nm indicating clustering of the nanoparticle.

In general, it was found that the copper oxide nanofluids can be quite stable for moderate time period without the presence of the surfactant, if the nanoparticle concentration is low. The time scale for non-sedimentation could vary from few days to months depending on the ultrasonication and stirring condition. Aggregation of ultrafine nanoparticle is inevitable in CuO nanofluid but the nanoscale cluster can still be stable in the mixture suspension.

Alumina (Al_2O_3) Nanofluids

Alumina nanoparticles are among the most widely used due to their abundance and low cost of mass production. The use of alumina nanoparticles in different base fluids has drawn considerable interest in applications including electronic cooling, deep drilling, thermal energy storage, etc. [187]. Beck [188] dispersed 20 nm diameter alumina nanoparticles in ethylene glycol with mass fraction ranging from 3.26 to 12.2%. The nanofluid was stabilized by ultrasonic mixing for several minutes and the suspension remained uniform during the experiments. Timofeeva [189] prepared nanofluids of alumina particles in water and ethylene glycol with three different particle size (11, 20, and 40 nm) and two different volume concentration (0.01% and 0.1%). The mixture was sonicated continuously for 5–20 h in an ultrasonic bath and highly agglomerated nanoparticles were observed in the experiments. It was found that particle with smaller diameter tends to form larger agglomerates and the agglomeration size increases with time as the sample ages. However, nanosamples were still found to be stable in both water and ethylene glycol. Esmaeilzadeh [190] prepared water-alumina nanofluid using 15 nm Al_2O_3 nanoparticle with 0.5 and 1% volume fractions. The mixture was stabilized through a 4 h process of ultrasonication and electromagnetic stirring. No sedimentation was observed throughout the testing period. Sarathi [191] dispersed 50 nm Al_2O_3 nanoparticles in distilled water by magnetic stirring for 3 h and ultrasonication for few hours. Sedimentation of particles was still observed after the sonication and stirrer was used during the experiment to minimize the sedimentation.

Use of surfactant and pH control can significantly enhance the stability of alumina nanofluid. Sharma [192] prepared stable water-Al_2O_3 nanofluid by mixing SDBS with one-tenth the mass of the nanoparticle (~47 nm) in the suspension. The nanofluid was observed to be stable for over a week if the volume concentration is less than 3%. With higher concentration, some sedimentation was observed. Teng [193] prepared water-Al_2O_3 fluid using 0.3 wt.% chitosan as the dispersing agent. The mixture with 0.5, 1.0, and 3.0 wt.% Al_2O_3 nanoparticles showed good suspension for 1 month during which the sample was placed statically. Jung [121] prepared water-based alumina nanofluid using a horn-type ultrasonic disrupter for 2 h. The nano-suspensions with 0–0.1 vol% Al_2O_3 nanoparticles (45 nm) were observed to be stable for more than 1 month with/without the addition of polyvinyl alcohol surfactant. Khairul made a more comprehensive study on the effects of surfactant toward the stability of Al_2O_3 nanofluid. He used different weight fractions from 0.05 to 0.2% of the dispersant SDBS to stabilize the water-Al_2O_3 (10 nm) nanofluid with nanoparticle weight ratio in the range of 0.05–0.15%. It was found that aggregation of nanoparticle still presents in the fluid, but 0.1% SDBS gives lowest mean aggregation size and maximum zeta potential which is an indication of good stability. Ho [194] prepared 0.1–4 vol.% water-Al_2O_3 nanofluid with the particle size of 33 nm. The nano-suspension was stable for at least 2 weeks after magnetic stirring for 2 h. and adjusting pH value to 3. Jacob [195] used similar method prepare stable suspensions of Al_2O_3 nanoparticles (~50 nm) in de-ionized water with 0.25, 0.5, and 1% volume. The mixture was stabilized by adjusting the pH value away from the isoelectric point and sonication for 5–6 h.

In general, it was found that the alumina nanofluids only exhibit short-term stability with mechanical stabilization methods. The stabilization period was enhanced to months if appropriate amount of dispersant was used.

Titanium Dioxide Nanofluids

Titanium dioxide is being widely used in various consumer goods and products including cosmetics, paints, dyes, plastics, drugs, etc. Nanoscale TiO_2 has high diffraction index and strong light scattering capability which makes them highly used in radiation protection productions, photocatalyst and photovoltaic [196–200]. The use of TiO_2 nanoparticle in nanofluid is promising due to its excellent chemical/physical stability and low cost from commercial manufacturers.

Ding and Wen [201] dispersed 30–40 nm TiO_2 nanoparticles in distilled water with 0.024% volume concentration. The stabilization of nanoparticles in water was realized by (1) cleaning of the bottles in ultrasonic bath; (2) adjusting the pH of the base liquid to pH = 3; (3) ultrasonification of the bottles containing dispersion for 15 min; and (4) shear mixing of the dispersion under the homogenizer for 30–180 min. The dispersion was found to be very stable for at least a couple of weeks without visually observable sedimentation. Murshed [202] prepared titanium dioxide nanofluid by dispersing TiO_2 nanoparticles in rod-shapes of $\varnothing 10$ nm × 40 nm (diameter by length) and in spherical shapes of $\varnothing 15$ nm in deionized water. The nanoparticles were dispersed uniformly using ultrasonic dismembrator for 8–10 h with/without 0.01–0.02 vol.% oleic acid and CTAB surfactants. It was found that nanoparticles agglomerated into large clusters without surfactant, and adding surfactant brought better stability which is indicated by the increment in nanofluid thermal conductivity. Turgut [203] prepared water-based TiO_2 nanofluid with particle size of 21 nm and particle volume concentration from 0.2 to 3%. The mixture was homogenized using ultrasonic vibration which breaks down the agglomerations. Yue-fan [204] prepared titanium dioxide colloidal suspension by dispersing TiO_2 nanoparticles (<20 nm) in transformer mineral oil with 0.003–0.05 g/L concentration. The particles were dispersed by just ultrasonic route and the suspension was stable for 24 h. He and Jin [205] prepared aqueous TiO_2 nanofluid by dispersing 20 nm diameter dry titanium dioxide nanoparticles in distilled water with 1.0, 2.5, and 4.9% mass concentration. The mixture was stabilized by first applying ultrasonication for 30 min, then processed in a medium-mill and finally adjusting the pH value to 11. The particle size distribution after each process stage was ~500 nm, ~120 nm, and ~95 nm while the nanofluids were found to be very stable for months. Charkraborty [206] prepared 0.1 wt.% water-based TiO_2 nanofluid using dry particles with size in the range of 30–50 nm. The nanofluid was homogenized by high shear mixer which breaks down the agglomerate and addition of 0.01 wt.% surfactant which ensures longer stability. Fedele [207] studied the characterization of water-based nanofluids containing TiO_2 (~72 nm) nanoparticles in mass concentrations ranging between 1 and 35%. The nanofluids were stabilized using 1–5 wt.% acetic acid as dispersant with 1 h sonication. The mean diameter of the static suspension decreases to around 51 nm after 35 days, indicating a partial precipitation. Such value returned to 76 nm after re-sonication for 1 h, suggesting the absence of further aggregation in the suspension. Said [208] dispersed 21 nm TiO_2 spherical particles in distilled water with 0.1% and 0.3% volume concentration. The homogenous dispersed solution was obtained after adding PEG 400 dispersant with two times the mass of the particles and passing through 30 cycles in a high-pressure homogenizer. No visual sign of aggregation and sedimentation was observed for a period of a month. Muthusamy [209] prepared stable titanium dioxide nanofluid by dispersing 50 nm diameter TiO_2 particles in ethylene glycol with 0.5, 1.0, and 1.5% volume concentration. The suspension was stabilized by merely mechanical stirring process and proved to be stable for more than 3 weeks with ~220 nm local clusters.

The titanium dioxide nanofluid exhibits relatively good stability (from few weeks to months). Different stabilization conditions were required for achieving optimum dispersion depending on the base fluid and particle characteristics.

Zinc Oxide Nanofluids

Zinc oxide is also among some of the widest used nanomaterials with its good electrical, electrochemical, and structural properties [210, 211]. ZnO nanoparticles exhibit in various form (particle, rod, thin film) and can be used in electroluminescent devices, chemical sensors, solar cells, etc. [212]. Zhang [213] prepared water-based zinc oxide nanofluid with 20 g/L concentration by dissolving ZnO nanoparticles (60–200 nm) in distilled water, sonicating for 30 min and milling for another 3 h. The average particle size increased slightly from 198.4 to 225.9 nm after being stored for 120 days, indicating good stability of the nanosuspension over time. Yu [214] dispersed dry ZnO nanoparticles (10–20 nm) in ethylene glycol with volume concentration ranging from 0.2 to 5%. The nanofluid was stirred and sonicated continuously for 15 min to 12 h. It was found that the average particle size decreases rapidly in the first 3 h and remained 210 nm afterward. The measured average particle size in the formulated nanofluids is much larger than the size of primary particles indicating ultrasonification was not able to break the agglomerates completely. Sagadevan [215] prepared ZnO nanoparticles (15–20 nm) first by solvothermal reaction and dispersed them in polyvinyl alcohol with magnetic stirring process and ultrasonic vibrator for 5 h. The dispersed mixture was clear and stable for up to 2 weeks. Esfe and Saedodin [216] prepared EG-ZnO nanofluid using 18 nm ZnO nanoparticles with volume concentration ranging from 0.25 to 5.0%. The suspensions were subjected to ultrasonic vibrator for 3–5 h in order to get a uniform dispersion and a stable suspension. Subramaniyan [104] prepared water-ZnO nanofluid by dispersing 0.1%–0.4 wt.% ZnO nanoparticles in water using ultrasonication for 20 min. It was found that ZnO nanofluids with 0.3 wt.% showed highest stability with the maximum zeta potential values. Visual sedimentation showed that 0.3 wt.% nanofluid is stable for 20–24 h without any trace of sedimentation but all the other fluids settle within 6–12 h. Raykar and Singh [105] synthesized water-soluble ZnO nanoparticle (non-spherical, 100–150 nm) via chemical precipitation method and dispersed them in deionized water. The mixture was sonicated for 1 h with the addition of acetylacetone as dispersant. The nanofluid was found to be stable over 9 months to 1 year. Suganthi and Rajan [106] prepared stable ZnO–water nanofluids with particle volume concentrations in the range of 0.25–2 vol.%. They dispersed ZnO nanoparticles (35–45 nm) into water with sodium hexametaphosphate (SHMP) surfactant under high shear homogenization for 20 min, followed by ultrasonication for 180 min. The high colloidal stability was verified by high absolute value of zeta potential as well as visual observation. Saliani [107] dispersed ZnO nanoparticles (4.45 nm) in glycerol with the aid of a magnetic stirrer. Ammonium citrate with the same mass of nanoparticles were used as a dispersant to enhance the stability of the nanofluids, and the suspensions were stable for at least several months with no sedimentation observed during the period.

It is noticed that the stability of zinc oxide nanofluids could be significantly enhanced by adding proper surfactant which could be potentially helpful for using them in the long-term application.

Iron Oxide Nanofluids

Iron oxide nanoparticles have attracted considerable interest due to their superparamagnetic properties and their potential biomedical applications arising from its biocompatibility and non-toxicity [217]. Asadzadeh [108] dispersed 0.05 vol% and 0.1 vol.% Fe_3O_4 nanoparticles (<50 nm) in ethylene glycol using vigorous mechanical agitation and ultrasonication for 1 h. The suspension was stable for 12 h without observable sedimentation. Sheikhabahai [109] prepared Fe_3O_4 nanofluid using EG–water mixture (50 vol.%-50 vol.%) with 0.02–0.1% particle volume loading. The 50 nm diameter Fe_3O_4 nanoparticles were added into the base fluid gradually under ultrasonic mixing for an hour. The nanofluid was stabilized with another hour of sonication and no sedimentation was observed for 8 h. Sundar [110] prepared water-based nanofluid using 36 nm Fe_3O_4 nanoparticles at 0.02, 0.1, 0.3, and 0.6% volume concentrations. The particles were uniformly dispersed in the base fluid at pH value of 3 with 2 h sonification. The uniform dispersion of the nanoparticles is established by visual observation for nanoparticle sedimentation and measuring the densities of nanofluid at different locations in the container. Župan and Renjo [111] prepared water-based ferrofluids by dispersing 50 nm diameter Fe_3O_4 nanoparticles in deionized water using ultrasonic bath for 90 min. The sonified colloid was stable for 1 h without dispersant or activating agent. However, visible sedimentation was observed in the bottom of the suspension after 24 h. Phuoc and Massoudi [218] dispersed Fe_2O_3 nanoparticles (20–40 nm) in deionized water with 0.2 wt.% Polyvinylpyrrolidone (PVP) or Poly(ethylene oxide) (PEO) as surfactant. The suspension was homogenized by magnetic mixing and ultrasonication for 30 min. It was observed that these nanofluids could remain stable for 2 weeks if the particle concentration is less than 2% and less than 1 week if the concentration is higher. Goshayeshi [219] prepared γ- and α-Fe_2O_3/Kerosene nanofluids with 2.0% volume concentration. The nanoparticles were added into the base fluids with oleic acid surfactant and stirred constantly, followed by 5 h sonication. The Fe_2O_3 nanoparticles could readily disperse in organic solvent and the suspension was stable for 10 days. Salari [220] prepared aqueous iron oxide nanofluids by dispersing 0.1–0.3 wt.% Fe_3O_4 nanoparticles (~20 nm) into the water using motorized magnetic stirrer with speed of 250 rpm for 30 min. The suspension was stabilized by adding 0.1 vol.% nonylphenol ethoxylate surfactant into DIW, ultrasonication for 30 min and adjusting pH values. The most stable nanofluid was obtained when pH = 8.43 and the suspension was stable for 25 days.

It can be seen from various studies that the iron oxide nanofluids exhibit relatively shorter stabilization period (less than a month) even with pH control and surfactant.

Silicon Dioxide Nanofluids

Silicon dioxide nanoparticles are of great interest in a variety of biomedical applications due to their stability, low toxicity and capability for functionalization with different molecules and polymers [221]. Fazeli [222] prepared water-SiO_2

nanofluids by dispersing 18 nm silica nanoparticles in distilled water with 3.5, 4, 4.5, and 5% volume concentration. The suspension was stabilized using ultrasonic bath for 90 min without any surfactant and the nanofluids were stable for a period of 72 h without any visible settlement. Jin [223] prepared 0.005–0.1% mass fraction silica nanofluid in mineral oil using 10–20 nm size SiO_2 nanoparticle. The particles were dispersed uniformly in the base fluid using magnetic stirring for 15 min and ultrasonication for 2 h. The 0.005% and 0.01% silica nanofluids were found to be stable for around 1 month, for the 0.02% silica nanofluid the stability of the suspension was reduced to 2 days, and the 0.1% silica nanofluid was stable for less than 24 h. Rafati [224] prepared silica nanofluid using a mixture of deionized water (75 vol.%) and ethylene glycol (25 vol.%) as the base fluid. SiO_2 nanoparticles with 14 nm average size were dispersed in the base fluid with 0.5, 1.0, and 1.5% volume concentration using ultrasonication. The nanofluids showed great stability even after 1 week. Noghrehabadi [225] dispersed 12 nm SiO_2 nanoparticles in water with 1% mass concentration using vertical mixer and ultrasonication for 60 min. The nanofluid was homogenized in ultrasonic bath every day to break down the agglomeration and minimize the sedimentation. Sharif [226] prepared polyalkylene glycol-SiO_2 nanolubricant by dispersing 30 nm SiO_2 nanoparticles in the base fluid using a magnetic stirrer for 1 h, and then surged using ultrasonic bath vibrator for 2 h. Minimum sedimentation was observed 1 month after the preparation of nanofluid with 0.2–1.5% volume concentration. Liu and Liao [227] prepared silica nanofluid in both water and alcohol. SiO_2 nanoparticles with 35 nm average diameter were dispersed in the base fluid with 0.2–2% mass concentration. The nanofluids were mixed with 0.5 vol.% SDBS surfactant and surged in super-sonic water bath for 12 h. The experimental results showed that the stability and uniformity of nanofluids were good at least in 1 month. Zhang [122] studied the influence of ultrasonication, dispersants, and pH on the stability of water-silica nanofluids. The nanofluids were prepared by dispersing 1.0 wt.% SiO_2 nanoparticles (~50 nm) in water with mechanical force agitation, ultrasonication, and addition of SDBS. It was found that the silica clusters were effective dispersed with average size of 63 nm in suspension under the sonication power of 500 W and sonication time of 120 min. The maximum absolute zeta potential was attained with SDBS concentration of 1.0% and pH value of 9.5. The good stability was also verified with long-term test in which the particle size remained unchanged after 7 days. Yang and Liu [114] prepared stable water-based nanofluid by dispersing surface-functionalized SiO_2 nanoparticles (30 nm) in water with 10% mass fraction. The nanofluid was kept stable for 12 months. Bagwe [228] prepared silica nanoparticles with different functional groups (including carboxylate, amine, amine/phosphonate, polyethylene glycol, octadecyl, and carboxylate/octadecyl groups) in water and studied their aggregation behavior in water. It was found that the nanoparticles prepared with appropriate amount of amine/phosphonate functional group were stable for more than 8 months in aqueous solution.

The silica-based nanofluids have relatively low stability using only physical stabilization method. The addition of surfactant has some marginal effect on the improvement of the stability, but surface modification could effectively make silica nanoparticles sustain much longer in the suspension environment.

Organic Nanofluids

Organic nanoparticles exhibit superior electric and thermal properties owing to their unique metal lattice or graphite structures, and have attracted attention for a wide range of applications in different fields. Dispersing carbon-based nanoparticles in liquid has not been as easy as other nanoparticle, as carbon-based particles have a strong tendency to agglomerate due to the strong intermolecular force. It has shown that pristine CNTs will precipitate rapidly in most of fluids even with prolonged sonication [229]. Consequently, surfactant and surface modifications have been used in almost all carbon-based nanofluid preparations.

Wen and Ding [230] prepared the stable aqueous suspension of MWCNT with 0–0.84% volume concentration. The prepared sample was stabilized following a sequence of steps involving: (1) ultrasonicating CNT sample in water bath for 36 h; (2) dispersing CNT in distill water with 20% by weight of sodium dodecyl benzene sulfonate (SDBS) with respect of CNTs; (3) ultrasonicating mixture in water bath for 24 h; (4) treating suspension with high-speed magnetic stirrer for 1 h. The aqueous CNT nanofluid was found to be very stable from months without sedimentation. Wusiman et al. [231] prepared MWCNTS in aqueous suspension with surfactant SDBS and sodium dodecyl sulfate (SDS). They changed the MWCNT concentration from 0.1 to 1 wt.%, with CNT/surfactant ratio varied from 1/1 to 4/1. The suspension was only subjected to ultrasonic mixing for 20 min and the samples with 3/1 CNT-surfactant ratio was found to be most stable for more than 1 month without sedimentation. SDBS was found slightly superior than SDS in their study with better thermal performance. Rashmi et al. [232] prepared aqueous dispersion of CNTs in the presence of gum arabic (GA), with concentrations of CNT and GA varying in the range of 0.01–0.1 wt.% and 0.25–5 wt.% respectively. The mixture was homogenized at 28,000 rpm for 10 min and further sonicated in water bath for 1–24 h. It was found that the optimum concentration of GA varies from 1.0 to 2.5 wt.% with increasing CNT concentration, and the nanofluid was found to be stable for more than 40 days. Quite few other surfactants have also been tested to show effective enhancement on the suspension stability of CNT nanofluid including polyvinylpyrrolidone (PVP) [233], hexadecyltrimethyl ammonium bromide (CTAB) [234], chitosan [235], and gemini surfactant [236]. The optimum concentration for each surfactant is dependent on its own property and the interaction with the carbon molecules.

Pre-treating CNTs in acid endows them with carboxylic acid and hydroxyl groups, which could effectively prevent the CNTs from aggregating over time [237]. Osorio et al. prepared functionalized CNTs by soaking CNTs in three different acid environments for 2 h: (1) $H_2SO_4/HNO_3/HCl$; (2) H_2SO_4/HNO_3; and (3) HNO_3. The treated CNTs were dispersed in water and the subsequent sedimentation over 20 days showed the good stability of the sample soaked in multi-acid environment. Zhang et al. [238] prepared water-soluble CNTs by the introduction of potassium carboxylate (−COOK) using potassium persulfate (KPS) as oxidant. The KPS-treated SWNTs was dispersed in deionized water with ultrasonic water bath and found to be stable over 1 month. Narisi et al. [239] prepared stable CNT nanofluid in water using a combination approach of surface functionalization, surfactant, and ultrasonication. The CNTs were first treated with KPS oxidant, and then dispersed in water with 0.25 wt.% SDS undergoing 45 mins' ultrasonication using both probe and bath ultrasonicator. The average particle diameter was examined using dynamic light scattering which remained constant (~200 nm) 2 months after preparation.

Although we have been discussing CNT-based nanofluid here, the concept and preparation method is quite similar for graphene/graphene oxide/fullerene-based nanofluid. Owing to their common surface properties, the dispersing method is widely acceptable between different types of carbon nanoparticles.

Special Nanofluid (High/Low Temperature)

For very high-temperature and low-temperature nanofluid, the preparation of nanofluid involves special steps as the mixing process cannot be performed in room environment. These nanofluids are usually based on materials which are not in liquid form at room temperature and atmospheric pressure (i.e., molten salt, liquified gas, low temperature refrigerant).

High-Temperature Nanofluid

For materials which are in the solid state at room environment, the preparation of nanofluid is usually performed by first dissolving the target material in a room temperature liquid (usually water), then dispersing nanoparticle in the solution, and finally evaporating water out and heating the composite to high temperature where it transformed into liquid. A typical example is the molten salt-based nanofluid which melts at temperature more than 200 °C. Shin and Banerjee [240] prepared silica nanofluids in alkali chloride eutectic. They first dissolve all chloride salt in distilled water, then dispersed 1.0 wt.% SiO_2 nanoparticle via ultrasonication bath for 100 min, and evaporated water in the vial on a hot plate at 200 °C until dried completely. The nanofluid showed good stability as particle size remained constant after repeated DSC cycling tests. Jo and Banerjee [241] prepared graphite nanofluid in molten carbonate salt using gum arabic as the surfactant. In his study, the surfactant and graphite nanoparticle were first dispersed in distilled water with 2 h sonication, then require amount of K_2CO_3 and Li_2CO_3 were dissolved in the suspension liquid with additional 3 h sonication. The final mixture was transferred to a petri dish and heated on a hot plate at 100 °C until fully dehydration. The nanomaterials showed good dispersibility with consistent thermophysical property measurements from repeated tests. Most other molten salt nanofluid preparations [242–246] have followed the same procedure used by Shin and Jo.

Low-Temperature Nanofluid

Most of the widely used refrigerants are in vapor state under room environment (i.e., R134a, R410a). Hence, preparing well-dispersed nanofluid using these materials is usually accomplished by first dispersing nanoparticle in a secondary fluid, and then putting the dispersed fluid into the refrigeration system before the refrigerant fills the test loop. Bi et al. [247] mixed TiO_2 nanoparticle into R134a by first dispersing the nanoparticle into mineral oil via conventional approaches, then put the mixture into the compressor to let the refrigerant mixing with the nanoparticle. Jwo et al. [248] followed a similar approach to mix Al_2O_3 nanoparticles in R134a using POE oil. Subramani and Prakash [249] prepared SUNISO 3GS oil-based nanolubricant with 0.06 wt.% Al_2O_3 nanoparticle which is stable for 3 days without coagulation or deposition. They then filled the nanolubricant in the compressor where it mixes with R134a.

In certain cases, nanoparticles can be added directly into the low temperature liquid as well. Anderson [250] dispersed MWCNT into liquid oxygen (LOX) by tipping the nanoparticles into the LOX gently and slowly. It is mentioned that great cares were taken at this point to avoid micro-scale boiling. The dispersion was then performed by ultrasonicating the mixture with a pre-cooled probe sonicator for 20 s. However, the study on cryogenic nanofluid is rather limited, which may be due to the inherent technical difficulty and narrow application field.

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