



Review Reproduction of Nanofluid Synthesis, Thermal Properties and Experiments in Engineering: A Research Paradigm Shift

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Abstract: The suspension of different nanoparticles into various conventional thermal fluids to synthesize nanofluids has been proven to possess superior thermal, optical, tribological, and convective properties, and the heat transfer performance over conventional thermal fluids. This task appears trivial but is complicated and significant to nanofluid synthesis and its subsequent utilization in diverse applications. The stability of mono and hybrid nanofluids is significantly related to stirring duration and speed; volume, density, and base fluid type; weight/volume concentration, density, nano-size, and type of mono or hybrid nanoparticles used; type and weight of surfactant used; and sonication time, frequency, mode, and amplitude. The effects of these parameters on stability consequently affect the thermal, optical, tribological, and convective properties, and the heat transfer performance of nanofluids in various applications, leading to divergent, inaccurate, and suspicious results. Disparities in results have inundated the public domain in this regard. Thus, this study utilized published works in the public domain to highlight the trend in mono or hybrid nanofluid formulation presently documented as the norm, with the possibility of changing the status quo. With the huge progress made in this research area in which a large quantum of different nanoparticles, base fluids, and surfactants have been deployed and more are still emerging in the application of these advanced thermal fluids in diverse areas, there is a need for conformity and better accuracy of results. Reproduction of results of stability, thermal, optical, tribological, anti-wear, and fuel properties; photothermal conversion; and supercooling, lubrication, engine, combustion, emission, thermo-hydraulic, and heat transfer performances of formulated mono or hybrid nanofluids are possible through the optimization and detailed documentation of applicable nanofluid preparation parameters (stirring time and speed, sonication duration, amplitude, mode, frequency, and surfactant concentration) employed in formulating mono or hybrid nanofluids. This proposed approach is expected to project a new frontier in nanofluid research and serve as a veritable working guide to the nanofluid research community.

Keywords: surfactant concentration; heat transfer performance; nanofluids; stability; sonication parameters; thermo-optical properties

1. Introduction

The advent of nanotechnology has brought about significant technological advancements in many fields of study. The birth of nanofluids as an advanced thermal transport media in the area of thermal management is a laudable and notable feat. Nanofluids (mono and hybrid nanofluids) have been extensively researched and established to be better than conventional thermal transport media due to their enhanced thermophysical and convective properties [1–11]. The application of diverse mono and hybrid nanofluids



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). to various thermal systems has been studied using experimental, analytical, and numerical approaches. Various types of convective heat transfer such as natural, mixed, and forced convection in laminar, turbulent, and transition regimes have been investigated by deploying nanofluids as thermal fluids against conventional thermal fluids [3,12–20]. Results have demonstrated an improvement in the heat transfer performance for the nanofluids at low concentrations compared with the conventional nanofluids. Moreover, pumping penalties for related studies showed a slight increase when nanofluids were used instead of conventional thermal fluids. In addition, the use of mono and hybrid nanofluids in heat transfer systems, such as radiators, refrigerators, microchannels, mini-channels, microtubes, heat pipes, solar collectors, etc., has been studied experimentally [10,21–33]. The suspension of different nanoparticles in the base fluids was revealed to enhance the overall heat transfer and flow performance of these thermal devices. Furthermore, mono and hybrid nanofluids/nanoparticles have been employed as coolants (in metal rolling process and metal machining operations) [34-39], lubricants (in the automobile) [40-43], thermal storage materials [44–46], sensors [47–49], drilling muds [50,51], chemically enhanced oil recovery material [52–54], desalination [55], solar applications [56,57], etc. These advanced thermal fluids revealed improved cooling, energetic and exergetic efficiency, thermal storage performance, surface finish, heat transfer and flow, and tribological characteristics, which are closely related to the enhanced properties of the base fluids due to nanoparticle suspension. Despite the laudable advantages associated with nanofluid deployment in various applications, the reproduction of such experiments and results is largely impossible.

Most of the literature in the public domain on nanofluid research does not give room for the reproduction of these experiments and results by not providing information or adequate variable data for the formulation of the studied mono and hybrid nanofluids. Thus, the repeatability of the experiments and results is not feasible. It can be inferred that the inability to reproduce the experiments due to a lack of detailed information for the formulation of the studied mono and hybrid nanofluids would definitely leave questions to be asked about the reliability and accuracy of the results obtained (thermo-optical properties and heat transfer characteristics) from such experiments.

The suspension of diverse nanoparticles into various base fluids to synthesize nanofluids has been proven to possess superior thermal properties to traditional thermal fluids. This exercise appears simple but complex in the true sense of it. Stability, which is the even distribution of mono and hybrid nanoparticles in the base fluid (in the absence or presence of surfactants), is key to the results associated with thermal and convective properties, and the performance of mono and hybrid nanofluids in various areas of application [58–66]. The stability of mono and hybrid nanofluids has been established to significantly affect their thermal-tribological-optical-fuel properties, lubrication, combustion, and convective heat performance [58,67–78]. Instability is marked by sedimentation and agglomeration of the mono and hybrid nanoparticles suspended in the base fluids. This consequently leads to inaccuracy and discrepancy in the results when the resultant mono and hybrid nanofluids are deployed in different applications [58,61,62,64,66,79–82]. This goes to show that obtaining adequate stability of mono and hybrid nanofluids is crucial. However, the stability of mono and hybrid nanofluids is strongly connected to parameters such as stirring time, rate, and temperature; sonication time, power, frequency, mode, and amplitude; and dispersion fraction (where surfactant is used) [5,70,74,76,83,84]. Sonication parameters (time, frequency, mode, probe tip size, and amplitude) are related to the sonication energy required to achieve homogenized and stable mono and hybrid nanofluids [72,77,85–88].

Parameters related to sonication, stirring, and dispersion fraction depend strongly on the volume, density, and base fluid type; weight/volume concentration, nano-size, density, and type of mono or hybrid nanoparticles used; type and weight of surfactant used, etc. Specific optimum values of the sonication parameters and dispersion fraction are required to achieve optimally stable mono and hybrid nanofluids. The stability of both mono and hybrid nanofluids has been demonstrated to affect their pH [80,89–91], thermal conductivity [91–94], viscosity [80,92,93], density [92,93], electrical conductivity [91], zeta potential values [77,90,95], aggregate size [96], sedimentation time and velocity [77,93,97], specific heat capacity [93], surfactant type and fraction [58,70,97], storage period [92], absorbency and extinction coefficient [80,89], light intensity and vapor generation [66], coefficient of heat transfer [58], and pumping power [98]. The majority of the literature in the public domain lack detailed information concerning the stirring and/or sonication parameters and/or dispersion fraction to reproduce stable mono and hybrid nanofluids and the results obtained for the studied thermal–optical–rheological properties and convective heat transfer performance in different applications. These scenarios have led to a wide disparity in the findings reported in the scientific literature concerning nanofluid studies. Convergence and reproductivity of these results are deemed possible only when relevant stirring and sonication parameters and dispersion fractions are determined and reported in the literature.

An exploration of the published works on mono and hybrid nanofluids in the public domain concerning their thermal, optical, tribological, anti-wear, and fuel properties; photothermal conversion; and supercooling, lubrication, engine, combustion, emission, thermo-hydraulic, and heat transfer performances in diverse applications (refrigeration and refrigerant; lubricants, coolants, solar collectors, automobile, thermal energy storage, nano-fuel, and engine performance; hot rolling and machining operations; mini-channel, microchannel, microtube, and mini-tube, etc.) is conducted with the aim of providing a discourse toward changing the present research paradigm on the preparation of nanofluids (mono and hybrid). This paper proposes a probable paradigm shift against the current approach of conducting experimental studies on mono and hybrid nanofluids. The provision of detailed data on the formulation parameters (mainly sonication, stirring, and deployment of surfactant) of mono and hybrid nanofluids is perceived to assist in shaping the future of nanofluid preparation toward accomplishing accurate, reliable, and reproducible experiments and results in terms of stability, thermal-optical-rheological properties, and thermal performance of these advanced thermal fluids in various applications. The outcome of this paper is expected to benefit the nanofluid research community. Figure 1 presents the temporal distribution of nanofluids and hybrid nanofluids as retrieved from the Scopus[®] database.



Figure 1. Temporal distribution of publications on nanofluids and hybrid nanofluids (Scopus: 20 May 2022).

This paper is divided into four sections. Section 1 is the general introduction of the discourse. Section 2 discusses nanofluid preparation and stability in relation to mono and hybrid nanofluids, while Section 3 addresses the scenarios observed over time concerning nanofluid (mono and hybrid) studies in terms of nanofluid preparation. Finally, the conclusion and future research outlook of mono and hybrid nanofluid research concerning the reproducibility of experiments and results are presented in Section 4. The schematic representation of this work is given in Figure 2 and Table 1.



Figure 2. Schematic diagram of this present work.

Year	Nanofluid	Concentration Application		Stirring Sonication		Surfactant	Sonicator Name/Type	Reference	Stability
2003	Al ₂ O ₃ /and CuO/DW	1–4 vol %	Property	-	12 h	NU	-	Das et al. [99]	Visual and density
2003	Al ₂ O ₃ /and CuO/DW	1–4 vol %	Natural convection	-	4 h	NU	-	Putra et al. [100]	Visual
2006	MWCNT, SiO ₂ , CuO, and fullerene/DIW, EG, and oil	0.01–0.5 wt %	Property	-	2 h	NU	-	Hwang et al. [101]	UV
2015	f-graphite/naphthenic oil	0.05–0.5 wt %	Property/nano- lubricant	1 h @ 2000 rpm and 50 °C	3 h @ 50 °C	NU	@ 150 W and 40 Hz	Lou et al. [21]	Visual
2016	Al ₂ O ₃ /water	0.1–1.5 wt %	Property	-	1 h	SDBS	-	Zawrah et al. [71]	pH and ZP
2016	$(60:40)$, SiO_x / EG-DIW ($60:40$), SiO_2 /DIW, and MgO/GL	1–6 vol %	Property	-	1–2 h	NU	@ 150 W and 40 kHz	Sharifpur et al. [7]	-
2007	CNT, Cu, Au, CNT-Cu (50:50), and CNT-Au (50:50)/DIW	varies	Property	-	1 h	Laurate salt	Bransonic [®] Ultrasonic Cleaner 1510	Jana et al. [102]	UV
2018	TiO ₂ -SiO ₂ , Al ₂ O ₃ -TiO ₂ , and Al ₂ O ₃ -SiO ₂ /polyalkylene glycol	0.02–0.1 vol %	Property/nano- lubricant	-	2 h	NU	-	Zawawi et al. [103]	Visual and UV
2019	CoFe ₂ O ₄ -SiO ₂ /W-EG (60:40)	0.1–1.5 vol %	Property/refrigerant	± 40 min	1 h	Carboxymethyl cellulose (0.1 mass ratio to NPs)	Hielscher (UP400St; 400W and 24 kHz)/probe	Safaei et al. [104]	UV and visual
2019	MWCNT, GNP, SiO ₂ , and Cu/DIW	0.057–2 vol %	Natural convection	30 min	30 min	SDS (4 mM)	-	Dixit and Pattamatta [105]	-
2018	Al ₂ O ₃ -MWCNT (90-95:10-5)/DIW	0.1 vol %	Natural convection	-	2 h	SDS	Hielscher (UP200S, 400 W and 50 Hz)/probe	Giwa et al. [16]	UV and viscosity
2017	Fe ₃ O ₄ and MWCNT/sulfinol-M	0.02–0.1 wt %	CO ₂ absorption	-	45 min	Triton X100 (weight ratio of 1:2)	@ 20 kHz and 400 W/probe	Nabipour et al. [106]	UV
2017	MWCNT and MWCNT-GNP/DW	0.075–0.25 wt % (MNFs) and 0.035 wt % (HNFs)	Forced convection in mini-tube	@ 65 °C	15 min (MNFs) and 2 h (HNFs) @ 18.4 kHz	PVP (1:1)	Telsonic Ultrasonics (SG-24-500P)/probe	Hussien et al. [23]	-

Table 1. Current scenario in nanofluid preparation.

Tabl	le 1.	Cont.

Year	Nanofluid	Concentration	Application	Stirring	Sonication	Surfactant	Sonicator Name/Type	Reference	Stability
2019	f-MWCNT, GNP, and f-MWCNT-GNP/diesel oil	0.05–0.5 wt %	Forced convection in a tube	30 min @ 5000 rpm (mixer, 330 W and 18000 rpm)	1 h	hexylamine and oleic acid	@ 750 W and 20 kHz/bath	Naddaf et al. [107]	UV
2019	MWCNT, Al ₂ O ₃ , and MWCNT-Al ₂ O ₃ /DIW	0.01 wt %	Corrugated plate heat exchanger	1 h (mechanical stirrer)	4 h	Span-80	-	Bhattad et al. [108]	Visual
2020	Al ₂ O ₃ -SiC (unmilled and milled)/EG-DIW (50:50 and 40:60)	0.4 and 0.8	Radiator (coolant)	1 h	4 h	-	-	Ramalingam et al. [25]	ZP
2020	MWCNT-GNP (1:1)/sea water	0.001–0.01 wt %	Solar evaporator	-	2 h	Gum Arabic	-	Ghafurian et al. [66]	Visual and ZP
2020	Fe ₃ O ₄ and MWCNT-Fe ₃ O ₄ /EG-water (20:80)	0.005–2 wt %	Photothermal conversion	-	2.5 h	Citric acid	-	Tong et al. [109]	Visual and ZP
2020	Cu/ and Cu-Gr/engine oil	0.03–0.6 wt %	Automobile nano-lubricants	4 h	-	Oleic acid	-	Ali et al. [42]	-
2020	Al ₂ O ₃ -GNP (85:15)/DIW	0.2–1.2 vol %	Turning of metals	-	6 h	-	Ultrasonication machine (model: 420 (100 W)	Khan et al. [110]	-
2020	SiO ₂ -GO (1:1)/DIW	0.04–0.2 wt %	Lapping operation (machining)	30 min @ 25 °C	90 min @ 5 s (on and off pulse)	-	Branson (S-450, 450 W)	Huang et al. [111]	-
2020	TiO ₂ , TiO ₂ -Ag, and β-cd-TiO ₂ -Ag/EG-DIW (40:60)	0.025–0.1 vol %	Thermal energy storage	30 min (mechanical stirrer)	1.5 h	-	Branson Ultrasonics	Li et al. [112]	ZP and TLAB dispersion analyzer
2020	MoS ₂ /, Al ₂ O ₃ /, and MoS ₂ -Al ₂ O ₃ /water	2 w%	Hot rolling lubrication	20 min @ 55 °C	30 min @ 50 °C	-	-	He et al. [36]	UV
2018	TiO ₂ /, Al ₂ O ₃ /, and ZnO/water	0.5–1 vol %	Forced convection in microchannel	-	30 min @ 21 °C and 1–2 cm	-	Optic Ivymen System, CY-500, 500 W, 20 kHz/probe	Topuz et al. [113]	Visual

Table 1. Cont.

Year	Nanofluid	Concentration	Application	Stirring	Sonication	Surfactant	Sonicator Name/Type	Reference	Stability
2020	MWCNT-Al ₂ O ₃ (4:1–1:4), MWCNT, and Al ₂ O ₃ /DIW	0.01 vol %	Forced convection in minichannel		6–8 h		Labman Scientific Instruments	Kumar and Sarkar [114]	pH modulation
2020	Al ₂ O ₃ -fly ash and SiO ₂ -fly ash/DIW	0.003–0.02 vol %	Microchannel with solar collector	-	120–130 min	Sodium oleate	E-chromTech (800 W, 20 kHz)	Thakur et al. [115]	ZP
2021	palm biodiesel (30 vol.) + diesel (70 vol.) + Al ₂ O ₃ or CNT or TiO ₂ NPs	-	Nano-fuel in diesel engine	35 min @ 2000 rpm	1 h	SDS (1:4 NP: SDS)	-	Fayaz et al. [116]	UV
2021	biodiesel (25 vol.), diesel (75 vol.), and HNPs (MWCNT-TiO ₂ ; 50–150 ppm)	-	Nano-fuel in diesel engine	1 h @ 60 °C	1 h (bath) and 20 min @ 15–30 Hz (probe)	Sorbitan oleate (2 vol %)	Qsonica (Q500, 500 W)/probe	Al-Hartomy et al. [117]	-

2. Nanofluid Preparation and Stability

2.1. Preparation Techniques

Mono and hybrid nanofluids were prepared by suspending mono nanoparticles (MNPs) and hybrid nanoparticles (HNPs) in orthodox thermal fluids (Figure 3). The stability of these thermal fluids is of utmost importance as it significantly influences their thermal–optical–rheological properties and thermal performance in different applications. Generally, mono and hybrid nanofluids are formulated using one-step and two-step strategies. The two-step method involves two processes; first, the synthesis of MNPs or HNPs, and second, suspending of the MNPs or HNPs into the base fluids. The two-step strategy is the most used method published in the literature for the formulation of mono and hybrid nanofluids. This method can be engaged in large-scale production of mono and hybrid nanofluids suitable for industrial application with cost benefits. The flaw associated with this method involves the possibility of sedimentation and agglomeration of MNPs and HNPs owing to the Van der Waals forces of attraction among the particles [118].



Figure 3. Representation of typical nanofluid preparation.

The one-step strategy involves simultaneous synthesis and suspension of MNPs and HNPs in the respective base fluids. This method is known to provide improved stability and homogeneity of mono and hybrid nanofluids, thereby eliminating arduous processes in comparison to the one-step strategy by reducing MNPs and HNPs clusters [118,119]. The industrial use of this method appears impracticable except for low vapor pressure fluids, and it is found to be expensive [120]. Various synthesizing techniques have been published in the open literature concerning the one-step strategy [119,121,122].

2.2. Stability

Suspending MNPs and HNPs in different base fluids induces charges leading to the formation of an electrical double layer (EDL) around the particle surface [123]. Thus, mono and hybrid nanofluids are said to be electrically conducting fluids. The imposition of a potential across these fluids causes oppositely charged electrodes to attract MNPs or HNPs and EDL. The formulation of EDL depends on the volume/weight fraction/concentration, size, and surface charge of the particles and the ion concentration in the base fluids. The application of mono and hybrid nanofluids strongly depends on the stability of these thermal fluids because their thermal–optical–rheological properties and thermal performance

are linked to the concentration of MNPs or HNPs in the suspension [124,125]. Under the two-step strategy, the agglomeration and sedimentation of mono and hybrid nanofluids can be avoided, thereby improving the stability of the same by employing mechanical (sonication), surfactant addition, surface modification, and pH control techniques. Figures 4–7 reveal the influence of viscosity, stability, thermal conductivity, and convective heat transfer performance, respectively, on sonication duration. In Figure 4, increasing sonication time is shown to enhance the stability (as measured using the zeta potential technique) of TiO_2 /water nanofluid. From Figure 5, the thermal conductivity of MWCNT/water nanofluid is noticed to enhance as temperature and sonication time increase. It is illustrated in Figure 6 that sonication time reduces nanofluid viscosity as the shear rate increases. In Figure 7, the peak convective heat transfer coefficient is achieved at the optimum sonication time under increasing flow development.



Figure 4. Effect of ultrasonic duration on stability of TiO₂/water nanofluid. Adapted from [95].



Figure 5. Effects of ultrasonication time and temperature on thermal conductivity of nanofluids [126].



Figure 6. Variation in dynamic viscosity as function of sonication time at various shear rates at 45 °C [126].



Figure 7. Influence of sonication duration on convective heat transfer coefficient (Re = 1200) of aqueous nanofluid. Adapted from [127].

With the aid of ultrasonicators, numerous studies have reported the effect of sonication time, amplitude, modes, and probe tip width on the thermal conductivity, absorbance wavelength, viscosity, cluster size, surfactants, diameter of CNTs, and particle size [72,77,78,80,86–88,128–130]. It is reported that optimum sonication parameters lead to optimum stability conditions and other relevant measured parameters. These findings indicate the need to optimize sonication parameters in relation to other parameters to achieve improved stability. Nevertheless, this is not the case for most studies reported in the literature for mono and hybrid nanofluids, irrespective of the purpose of the experiments. Surfactants are chemical compounds used to improve the stability of mono and hybrid nanofluids by reducing the electrostatic repulsion and Van der Waals interaction between particles to evade the agglomeration of particles in the base fluid [131]. An increase in thermal conductivity, zeta potential, surface tension, and viscosity of mono and hybrid nanofluids under the utilization of different surfactants has been published in the literature [121,124,129,132–134]. In addition, the pH can be modulated to improve the stability of mono and hybrid nanofluids. The surface electric charges introduced due to the suspension of MNPs or HNPs into base fluids are altered to enhance the stability of mono and hybrid nanofluids. The farther the pH value from the isoelectric point (IEP), the greater the stability of the mono and hybrid nanofluids. Furthermore, the surface modification technique can be used to improve the stability of mono and hybrid nanofluids. It is worth noting that this technique is always surfactant-free with more improved stability [125,128,135]. The effect of surfactants on the thermal conductivity and thermal efficiency of mono and hybrid nanofluids is shown in Figures 8 and 9, respectively. From Figure 8, variation in the thermal conductivity is observed for the 0.5 wt % MWCNT/DW nanofluid formulated using different surfactants and subjected to increasing temperature. Figure 9 shows that the thermal efficiency of a hybrid nanofluid varies with the type and concentration of surfactant used in the formulation. For all the hybrid nanofluids, the thermal efficiency increased as the Reynolds number rises.



Figure 8. Influence of different surfactants (GA, SDBS, and SDS) thermal conductivity of aqueous MWCNT nanofluids [126].



Figure 9. Influence of surfactants (PVP and SDS) on thermal efficiency of hybrid nanofluids in a helical coil heat exchanger [58].

For the characterization of mono and hybrid nanofluids, numerous techniques have been reported in the open literature. These include Raman spectroscopy, Fourier transform infrared spectroscopy, high-resolution transmission electron microscopy, X-ray diffractometer, scanning electron microscopy, vibrating sample magnetometer, transmission electron microscopy, light scattering, and energy-dispersive X-ray spectroscopy [1,77,136–141]. Scanning electron microscopy is the most reported technique for characterizing mono and hybrid nanofluids. To monitor the stability of mono and hybrid nanofluids, visual inspection, zeta potential, ultraviolet-visible (UV) spectrophotometer, and thermal property tracking (viscosity, thermal conductivity, turbidity, and density) methods are engaged [12,75,76,132,137,142–145].

3. Scenarios on Formulation and Stability of Nanofluids

3.1. Classical Scenario on Formulation and Stability of Nanofluids

After over twenty-five years of research on nanofluids as advanced thermal fluids, an attempt to examine the literature on the trends related to the formulation and stability characteristics of mono and hybrid nanofluids was carried out. The scenarios generally portrayed by the publications in the open literature concerning the preparation of nanofluids are presented in Figure 10. Under the classical scenario of nanofluid formulation and stability characteristics, cases of no sonication and stirring parameters, no surfactant concentration details, and no visual inspection of stability or mention of stability are portrayed. Publications from pioneering studies down to present works on the thermal properties and applications of mono and hybrid nanofluids are considered with the aim of pinpointing the shortcomings related to this scenario and, by extension, nanofluid preparation and stability.



Figure 10. Scenarios portrayed by nanofluid preparation studies.

The hydraulic and convective heat transfer behavior of aqueous Cu nanofluid with a volume fraction of 0.3-2% flowing in a tube under turbulent conditions was examined [146]. The authors did not provide information concerning the formulation (sonication and stirring parameters) and stability of the Cu/water nanofluid used in their study. The thermal conductivity of DIW and EG-based Al₂O₃ and CuO nanofluids with a volume fraction of 1-5% was measured [147]. It was reported that after dispersing the NPs of Al₂O₃ and CuO in the respective base fluids, they were thoroughly shaken to homogeneity in a mixing chamber made of polyethylene without the provision of rate and duration of mixing. In the work of Eastman et al., in which the thermal conductivity of water-based Cu nanofluid (up to 0.5% volume fraction) + thioglycolic acid (< 1 vol %) was experimentally determined, no report of stirring or sonication was provided, except mentioning the dispersion of the MNPs in the EG [148].

The measurement of the viscosity and thermal conductivity of Fe/water (up to 2.93%) and Fe₃O₄/DW (1–5%) nanofluids under varying magnetic field strengths and directions was reported [149]. An ultrasonic vibrator was engaged in the preparation for undisclosed hours to enhance the stability of Fe and Fe₃O₄ NPs in the respective base fluids using oleic acid and sodium dodecyl benzenesulfonate as surface activators, respectively. The

authors did not disclose the weight or proportion of the different NPs to the activators used in the study. Mahrood et al. did not provide details of the mechanical stirring and ultrasonication deployed to formulate DW + 0.5 wt % CMC-based Al_2O_3 and TiO_2 nanofluids (0.1–1.5 vol %) utilized in a vertical cylinder as coolants to study their free convection heat transfer behavior [150]. In addition, the ultrasonication of Al_2O_3 nanofluids (0.85–2.95 wt %) used for the free convection heat transfer characteristics of these fluids contained in a vertical cylinder exposed to a constant heat flux from the top wall was reported [151]. However, the ultrasonication parameters are not presented in this work.

Owing to research progress in this field, the hybridization of NPs was deployed to improve both the thermo-physical properties and thermal performance of traditional thermal fluids and MNFs. The thermal properties of DIW and EG-based Ag, Cu, Al, Al₂Cu (70:30), and AgAl₂ (30:70) nanofluids with concentrations of 0.2–1.5 vol % were determined [152]. Oleic acid (1 vol %) as a surfactant in addition to stirring and ultrasonication was used to improve the stability of both MNFs and HNFs. However, the parameters involved in the sonication and stirring, especially the duration, are not documented in this work. No stability test was conducted, despite the authors emphasizing the crucial importance of stability to efficient heat transfer application of the prepared samples. The thermal conductivity of EG-W (40:60)-based CuO-SWCNT (50:50) nano-lubricants with volume fractions of 0.02–0.75% under an increasing temperature of 20–50 °C was determined [153]. No information concerning the preparation parameters and stability of the HNFs is provided.

Using a two-step strategy, the viscosity of water-based GNP, SiO₂, and GNP-SiO₂ nanofluids (with volume fractions of 0.05–1%) under increasing temperature (25–50 °C) was measured [154]. The authors reported the use of a magnetic stirrer and ultrasonication to reduce the agglomeration of the NPs and thus improve the stability of the MNFs and HNFs without specifying the related values of the preparation parameters. However, the stability was checked using zeta potential (ZP). In the work of Sahoo and Kumar, though a magnetic stirrer and ultrasonication were employed to homogenize and stabilize the formation of Al_2O_3 -TiO₂-CuO (33:33:33)/water nanofluids (volume fractions of 0.0125–0.1%) used for the measurement of the viscosity at 35–50 °C, the values of these preparation parameters are not specified. Stability was evaluated based on visual inspection [155].

The heat transfer performance of ammonia–water (20:80) + Al_2O_3 NPs + Triton X110 as a nano-refrigerant was examined [156]. The preparation of this advanced thermal fluid involved ultrasonic agitation for better and more even dispersion of the NPs. The authors failed to provide the detailed procedure and preparation parameters related to the nano-refrigerant preparation. A two-step strategy was used to prepare six samples of DW-based Ag and SWCNT nanofluids (0.1–0.3 vol %) to investigate the thermo-physical properties (viscosity and thermal conductivity) of the MNFs and their heat transfer performance as secondary working fluids in a refrigerating system [157]. Intensive magnetic stirring and sonication performed are not documented. A two-process method was engaged to formulate the DW-based Al + Al_2O_3 nanofluids (volume fraction of 1–5%) deployed as working fluids in an evacuated tube solar collector studied for the thermal heat transfer performance [158]. The ultrasonic mixing (at 25–30 °C) to break the HNPs for even distribution and enhancement of stability was reported. The values involved in the ultrasonic mixing are missing in the publication.

The heat transfer performance of water-based rGO, GO, rGO-CNT (3:1), rGO-CNF (2:1), and rGO-GNP (1:1) nanofluid (0.05 wt %) in a constant heat flux-heated horizontal tube under turbulent conditions was investigated [159]. No report of the preparation procedure or stability test was provided by the authors. The thermal transfer performance of Al_2O_3 -Ag (97.5:2.5)/DW nanofluids (0.2 vol %) as nano-coolants in a constant-temperature helical coil heat exchanger using PVP and SDS (0.1–0.4 wt %) as surfactants was investigated [58]. The work failed to provide parameters involved in the preparation of the hybrid nano-coolants. In an attempt to study the heat transfer performance of water-EG (50:50)-based Ag-TiO₂ (0.1–0.3%), TiO₂, and Cu-TiO₂ (0.1%) nanofluids as coolants in a radi-

ator, the nanofluids were formulated by exposing them to ultrasonication and maintaining pH 7 using ammonium hydroxide solution [62]. The detailed preparation procedure is not given. Fe_3O_4 -TiO₂/DIW nanofluids for the solar-enabled separation, purification, and photothermal characteristics were studied [160]. However, the authors failed to report the formulation of the HNFs in terms of preparation parameters related to the possible stirring and ultrasonication of the HNPs suspended in DIW.

Ali and Xianjun reported no preparation details about the formulation of an engine oil (98 wt %)-based nano-lubricant (Al₂O₃ (0.05%) + TiO₂ (0.05%) + oleic acid (1.9%)) employed to study the thermal stability and heat transfer behavior of the nano-lubricant for vehicular use [43]. Kiani and co-workers reported the use of the ultrasonication process to homogenize water-based Al₂O₃, CuO, and AgO nanofluids (with a volume fraction of 1–4%) studied as coolants for the thermal management of lithium ion batteries as a thermal storage device [161]. However, they failed to report the values of the sonication parameters involved in the preparation procedure which can help in replicating the study. To improve the performance of eco-friendly, minimum-quality liquid techniques in grinding operations, Rabiei et al. employed nanofluids (MNFs and HNF) and ultrasonic-assisted grinding [162]. The work did not publish any information on how the studied waterbased MNFs (0.25 wt % Al₂O₃ and MWCNT) and HNF (0.25 wt % Al₂O₃-MWCNT) were prepared, thus making the reproduction of this work impossible. The tribological properties of 0.5 wt % lanthanum trifluoride–graphene oxide/paraffin as a hybrid nano-lubricant were studied [163]. Oleic acid was used to modify the surface of lanthanum trifluoride– graphene oxide NPs. The preparation details of how the HNPs were dispersed in paraffin to formulate a stable hybrid nano-lubricant are missing.

The heat transfer performance and entropy generation capability of aqueous GNP- Al_2O_3 (50:50) nanofluid (0.1% volume fraction) flowing in a minichannel heat exchanger (two-pass multiport) incorporated with a thermoelectric cooler under laminar conditions was investigated [164]. The authors only stated the deployment of ultrasonication in preparing the HNF without using a surfactant to stabilize it. However, they failed to report the ultrasonication values related to the nanofluid formulation. Based on this, reproducing this study would be difficult. Moreover, the convective heat transfer characteristics of DIW-based TiO₂-Al₂O₃ nanofluids (volume fraction of 0.1–1%) flowing in fan-shaped and rectangular microchannels under laminar conditions was studied [165]. Using a two-step process, mechanical and ultrasonic disruption of the nanofluids with PVP as a surfactant was reported without giving details of the parameters related to the same. The reviewed studies on the preparation and stability of nanofluids under this scenario show that the absence of preparation parameters (ultrasonication and stirring values) would make the reproduction of these studies difficult. To obtain similar results in terms of the nanofluid stability, properties, and thermal performance, authors must provide adequate preparation details that would facilitate the repetition of their studies. It is pertinent to mention that most nanofluid studies in the literature did not give adequate preparation details that would encourage the reproduction of their studies. This is not an ideal way to conduct experimental studies and needs to be reconsidered.

3.2. Contemporary Scenario on Formulation and stability of Nanofluids

By reporting some specific sonication and stirring parameters and surfactant concentrations engaged in nanofluid preparation, another scenario is observed in the public domain concerning nanofluid studies. Bearing in mind the need for and importance of sufficient dispersion of NPs in base fluids, and in comparison with the work of [147], where no effort was made to break up NPs of Al₂O₃ and CuO, Das and co-workers employed an ultrasonic vibrator for 12 h to improve the dispersion of Al₂O₃ and CuO in DW [99]. This preparation was conducted prior to measuring the thermal conductivity of the resulting MNFs with volume fractions of 1–4% under different temperatures (21–51 °C). Using visual inspection and density checks, the homogeneity of all samples of nanofluids was confirmed. A slightly higher thermal conductivity was observed in this study compared to that of [147], mainly due to the further breakdown of the agglomerated MNPs. However, the details of the ultrasonic vibrator parameters are not provided, which will hinder the reproduction of this work. To measure the thermal conductivity, viscosity, and rheological behavior and to investigate the free convection heat transfer performance of MNFs in a horizontal cylinder under thermal constraint, Putra and co-workers performed ultrasonic vibration (4 h) to improve the dispersion of Al₂O₃ and CuO particles in DW without a surfactant [100]. No sedimentation was observed after 6 h of preparation. Also lacking are the values of the ultrasonic vibrator parameters required to reproduce this experiment, and thus, no reproduction of this work is possible. The lubrication and thermal conductivity performance of DIW, EG, and mineral oil-based MWCNT, SiO₂, CuO, and fullerene nanofluids were carried out, in which their stability was measured using a UV–Vis spectrophotometer, and the breaking of agglomerated NPs was executed using a vibration disruptor for 2 h, without any details of the other sonication parameters [101].

The reliability, efficiency, and heat transfer behavior of surface-modified graphite/naphthenic oil nano-lubricant (with mass fractions of 0.05–0.5%) used in a refrigerating system were investigated [21]. The work demonstrated that the nano-lubricants were prepared by stirring the samples at 2000 r/min for 60 min and 50 °C. To further homogenize and stabilize the samples, ultrasonic homogenization was performed for a period of 3 h (at 50 $^{\circ}$ C), 1 h each at an interval of 3 h. Lacking in this work are the values of the sonication parameters of the amplitude, mode, and frequency to aid the reproduction of the studied nano-lubricants. The stability and electrical conductivity of Al_2O_3 /water nanofluid with 0.1–1.5 wt % were examined [71]. These samples were prepared by optimizing the amount of SDBS used as a surfactant, ultrasonicating the mixture for 1 h, and stabilizing the mixture at pH 8. ZP was used to check the stability of SDBS + Al_2O_3 /water nanofluid. This detail provided by the authors is not sufficient for reproducing the samples and study. Sharifpur and co-authors prepared CuO/GL, SiO_x/EG-DIW (60:40), SiO₂/DIW, and MgO/GL nanofluids (volume fractions of 1–6%) by stirring and sonication (for 1–2 h) to homogenize and stabilize prior to measuring the density of the nanofluid samples [7]. These preparation details are found to be insufficient for reproducing the experiment.

In the work of Jana and co-workers, the thermal conductivity of DIW-based CNT, Cu, Au, CNT-Cu (50:50), and CNT-Au (50:50) nanofluids was measured [102]. Ultrasonication of the samples was performed for 1 h, with an absorbency test to check their stability. The sonication parameters outside the duration are not reported. The viscosity and thermal conductivity of polyalkylene glycol-based TiO₂-SiO₂, Al₂O₃-TiO₂, and Al₂O₃-SiO₂ nanofluids (0.02–0.1 vol %) at 30–80 °C were measured, in which no information was given regarding the sonication characteristics except optimizing the sonication time (2 h) [103]. Both visual inspection and UV spectrophotometry were engaged to check the stability of the hybrid nano-lubricants. The thermal conductivity of antifreeze (W-EG (60:40))-based CoFe₂O₄-SiO₂ nanofluid (with a mass fraction of 0.1%–1.5% and at 25–50 °C) to be used in refrigeration condensers was measured [104]. Carboxymethyl cellulose (as a surfactant) with a mass ratio of 0.1 to the HNPs was utilized in the preparation of the nanofluid to improve its stability. The mixture was stirred for 40 min and ultrasonicated for 1 h to break down the HNP clusters and thorough saturation of the surfactant. Absorbance was measured to evaluate the stability of the sample in addition to visual inspection. However, stirring speed and other ultrasonication values are missing to enable the reproduction of the experiments and results.

The free convection thermal transfer performance of DIW-based MWCNT, GNP, SiO₂, and Cu nanofluids (volume fraction of 0.057–0.2%) contained in a square enclosure exposed to magnetic excitation (0.13 T and 0.3 T) was investigated [105]. Using a two-step process, the MNFs were prepared by engaging SDS as a surfactant to improve their stability. The corresponding mixture was stirred for 30 min and ultrasonicated for another 30 min for homogeneity. The rate of stirring and ultrasonication amplitude, pulse, and frequency to assist the reproduction of the experiment are lacking. To study the free convection thermal behavior of aqueous Al_2O_3 -MWCNT (90-95:10-5) nanofluids (0.1 vol %) in a square

enclosure, Giwa and co-workers [16] engaged the two-step strategy by sonicating the HNP, DIW, and SDS (as a surfactant) for 2 h. Reproducing this experiment is not likely as the other parameters related to sonication and surfactant concentration are not provided.

The absorption (equilibrium and rate of absorption) of CO₂ using sulfinol-M-based Fe₃O₄ and MWCNT nanofluids with a concentration of 0.02–0.1 wt % under pressure was studied [106]. Without stirring, ultrasonication of NPs (MWCNT and Fe₃O₄) + Triton X100 (weight ratio of 1:2) + sulfinol-M was performed for 45 min. Sonication parameters (amplitude, frequency, and pulse) outside the duration are not provided to aid the reproduction of the work. The hydraulic and thermal performance of DW-based MWCNT and MWCNT-GNP nanofluids in a mini-tube subjected to a uniform heat flux under laminar flow conditions was examined [23]. Stable MNFs and HNFs were prepared by stirring PVP + DW at 65 °C and sonicating MWCNT + PVP (1:1) + DW (0.075–0.25 wt %) for 15 min and MWCNT + PVP + DW + GNP (0.035 wt %) for 2 h, both at a sonication frequency of 18.4 kHz. A similar preparation procedure was reported by [22] when the heat transfer and entropy generation performance of the same MNFs and HNFs as that of [23] were investigated in a constant heat flux microtube under laminar flow. However, the stirring speed and amplitude of sonication involved in these studies are not provided for the reproduction of the results and experiments.

The heat transfer performance of diesel oil-based functionalized MWCNT, GNP, and MWCNT-GNP (1:1) nanofluids (0.05–0.5 wt %) at different flow rates (18.75–50 mL/s) in a constant heat flux straight pipe subjected to laminar flow was investigated [107]. Both hexylamine and oleic acid were used to functionalize the NPs. To prepare and improve the stability of the MNFs and HNFs, mixing was carried out using a shear mixer at 5000 rpm for 30 min and providing ultrasonication of the test samples for 1 h. UV spectrophotometry was used to check the stability of the tested samples. Sonication parameters apart from the duration are not provided to aid the reproduction of the study and findings. Using a two-step strategy of preparation, the exergetic and energetic performance of DIWbased MWCNT, Al₂O₃, and MWCNT-Al₂O₃ (4:1–1:4) nanofluids (with a concentration of 0.01 wt %) in a corrugated plate heat exchanger (counterflow type) was examined [108]. Stirring (mechanical stirrer) was provided for 1 h and ultrasonication was performed for 4 h to aid homogenization of the MNFs and HNFs with the addition of Span-80 as a surfactant. Details of the stirring and sonication are missing in the published work and this will not enable the repetition of the experiment and results. The work of Ramalingam and co-workers showed that EG-DIW (50:50 and 40:60 w/w)-based Al₂O₃-SiC (unmilled and milled) nanofluids (0.4 vol % and 0.8 vol %) deployed as coolants in a radiator for the investigation of the thermal performance were formulated by stirring for 1 h and sonicating for 4 h. Stability was tested using the ZP technique [25]. The stirring and ultrasonication parameters (amplitude and frequency) provided in the study are not sufficient to reproduce the results and experiments.

The solar evaporation efficiency of seawater-based MWCNT-GNP (1:1) nanofluids (0.001–0.01 wt %) under the influence of sonication duration (30–240 min) was examined [66]. The HNFs were prepared using gum arabic as a surfactant and ultrasonicating the mixture for 120 min as the optimum sonication duration. This information is not adequate for reproducing this experiment. Tong and co-workers reportedly prepared EG-water (20:80)-based Fe₃O₄ and MWCNT-Fe₃O₄ (1:4–4:1 mixing ratio and 0.005–2 wt %) nanofluids for the investigation of the thermo-optical properties and photothermal energy conversion characteristics [109]. With citric acid as a surfactant, the mixture was ultrasonicated for 2.5 h, and ZP and visual observation were used to evaluate their stability. The nanofluid preparation parameters of sonication duration provided in the work are not adequate to reproduce this study and its findings.

The anti-wear properties of automobile sliding interface using mono and hybrid nanolubricants (Cu/ and Cu-Gr/engine oil + OA (wt %) nanofluid with 0.03–0.6 wt % were investigated [42]. It was reported that the nano-lubricants were formulated by stirring for 4 h without giving the speed involved and hence not providing sufficient detail to replicate the study. The use of an ultrasonication machine for 6 h homogenization to prepare DIW-based Al₂O₃-GNP (85:15) nanofluids (0.2–1.2 vol %) as coolants for sustainable evaluation of machine turning of a cobalt-based superalloy (Co-20Cr-15W-10Ni) using the prepared hybrid nano-lubricants was reported [110]. Reproducing this study is not possible as the preparation parameters published are not sufficient to carry out this experiment. In studying the mechanism and lapping operation performance of SiO_2 -GO (1:1)/DIW nanofluids (0.04–0.2 wt %), Huang and co-authors prepared the samples by first suspending GO NPs in DIW ultrasonically for 30 min, then stirring SiO₂ NPs and GO nanofluid for 30 min at 25 °C and finally sonicating the mixture for 60 min (under 5 s pulse on and off) [111]. The stirring speed and sonication amplitude and frequency to reproducible the work are not reported. To save energy, eco-friendly cold thermal energy storage media of EG-DIW (40:60)-based TiO₂, TiO₂-Ag, and β -cd-TiO₂-Ag nanofluids (0.025–0.1 vol %) were prepared and investigated for the thermal conductivity, stability, and supercooling characteristics [112]. Using the two-step method, the samples were prepared by mechanically stirring for 30 min and ultrasonicating for 1.5 h. To reproduce this work, both the stirring rate and the ultrasonication amplitude and frequency are required to aid the reproduction of the experiment and findings.

In the hot strip rolling of E235B steel, the lubrication performance of 2 wt % MoS₂/, Al_2O_3 /, and $MoS_2-Al_2O_3$ /water nanofluids as nano-lubricants/coolants was investigated [36]. The two-step preparation process involved stirring at 55 °C for 20 min and ultrasonicated at 50 °C for 30 min. These parameters are not sufficient to repeat the experiment and obtain the same results. The thermo-hydraulic performance of $TiO_2/$, $Al_2O_3/$, and ZnO/waternanofluids (0.5–1 vol %) in a constant surface temperature circular microchannel (horizontal) under laminar flow conditions was studied [113]. The MNFs were formulated by ultrasonic disruption of the samples for 30 min at 21 °C using a probe height of 1–2 cm. These parameters are found not to be adequate to reproduce the experiment and findings. The two-step process was engaged to prepare 0.01 vol % DIW-based MWCNT-Al₂O₃ (4:1–1:4), MWCNT, and Al₂O₃ nanofluids utilized for the investigation of the heat transfer performance of these fluids in rectangular mini-channels [114]. Homogenization of the samples was performed in an ultrasonicator for 6-8 h, which is not sufficient to be used to reproduce this work and result. The thermal, entropy generation, and exergy performance of DW-based Al_2O_3 -fly ash (1:4-4:1) and SiO₂-fly ash (1:4-4:1) nanofluids (with volume fractions of 0.003-0.02%) flowing inside a microchannel incorporated direct absorption solar collector under laminar flow conditions were studied [115]. The preparation of these HNFs involved the use of sodium oleate as a surfactant and ultrasonication for 120–130 min. More sonication details (amplitude, frequency, and pulse) are required to reproduce the work.

The fuel properties, engine performance, and emission behavior of nano-fuel (palm biodiesel (30 vol.) + diesel (70 vol.) + Al_2O_3 or CNT or TiO₂ NPs + SDS) in a single-cylinder diesel engine were investigated [116]. SDS was used as a surfactant to improve the stability of the nano-fuel. To produce a stable nano-fuel, an NP:surfactant ratio of 1:4, stirring at 2000 rpm for 35 min, and ultrasonication for 1 h were implemented. Stability was monitored using an absorbency test. Other sonication parameters outside the duration reported are missing to enable reproductivity of this experiment and findings. The combined influence of Phoenix dactylifera biodiesel (25 vol.), diesel (75 vol.), and HNPs (MWCNT-TiO₂; 50–150 ppm) on the combustion, performance, and emission characteristics of a single-cylinder diesel engine was investigated [117]. The two-step method was engaged to prepare the nano-fuel with the use of sorbitan oleate (2 vol %) as a surfactant to avoid agglomeration and improve stability. The magnetic stirring of the mixture (at 60 °C) lasted for 1 h, followed by bath sonication for 1 h and finally probe sonication for 20 min at 15–30 Hz. The stirring rate and details of other sonication parameters (amplitude and mode of sonication) are required to reproduce this experiment.

In the formulation of biodiesel blends with diesel and NiO nanoparticles using diesterol as a surfactant, magnetic stirring was performed for 60 min, followed by ultrasonication for 75 min to homogenize the mixture. Details of the sonication parameters (amplitude and frequency) and stirring rate are not given to reproduce the experiment [166]. To study the influence of Sr/ZnO nanoparticles on the engine performance characteristics of Ricinus communis biodiesel–diesel, a two-step strategy was deployed to prepare the mixture with the use of sonication for even distribution of the same [167]. The parameters related to the sonication process are not provided to enable the reproduction of the experiment.

3.3. Future Scenario on Formulation and Stability of Nanofluids

In the context of reproducing experiments and results, a future scenario as suggested by this present study represents a trend in nanofluid studies in which the nanofluid preparation involves the provision of relevant parameters sufficient to reproduce both the study and findings. This scenario describes a situation in which the preparation parameters are adequate to reproduce the experiment and results in question. According to the literature in the public domain, few studies have been published in this regard, and this could be foreseen as the future of nanofluid studies where accurate and reliable results can be achieved, coupled with reproducibility of the experiments and results. The hydraulic and heat transfer performance of water-based Al₂O₃ and TiO₂ nanofluids with a volume concentration of 1–10 vol % flowing in a circular pipe under turbulent conditions for the first time was studied [168]. Stable MNFs of Al_2O_3 and TiO_2 at pH of 3 and 10 were formulated using a mixer operated at 10000 rpm for 2 h. With sufficient information concerning the preparation of the nanofluids, this result is reproducible. The stability of the MNFs was monitored using visual inspection and density measurement. The preparation of DW and paraffin-based Al_2O_3 and WO_3 nanofluids (with a mass fraction of 0.1–5%) was examined for viscosity (at 5–65 °C) and rheological behavior (2.6–64.6 s⁻¹) [169]. Stirring was performed at 500 rpm for 30 min while ultrasonication was carried out at an amplitude of 60% and on-pulse of 0.5 s for 60 min with 5 min rest after each 20 min. Stability was established using ZP. These preparation parameters are sufficient to reproduce the study. To study the viscosity, electrical conductivity, and stability of MWCNT-Fe₂O₃ (20:80)/DIW nanofluids (0.1–1.5 vol %), Giwa and co-workers used a two-step process to prepare the HNFs with the use of SDS as a surfactant to enhance stability [81]. Optimal values of 0.5 (dispersion fraction), 120 min (sonication time), and 70% (sonication frequency and amplitude) were reported, which could be used to reproduce this work. Stability was checked by measuring the absorbance and viscosity of the HNFs.

The viscosity of Al₂O₃ (20, 80, and 100 nm)/glycerol nanofluids with a volume fraction of 1–5% was measured after preparing the MNFs using a two-step approach [72]. Without the use of surfactant, the mixture was ultrasonicated for 6 h (20–30 nm samples) and 3 h (80 and 100 nm samples) at a sonication amplitude of 75% with 0.8 s (pulse on) and 0.2 s (pulse off). To measure similar properties as those in the work of [81], Ref. [75] prepared 0.1 vol % Al₂O₃-MWCNT (90:10–20:80)/DIW nanofluids via a two-step process by ultrasonicating for 2 h at a frequency of 70% and amplitude of 75% with SDS dispersion fraction of 1. Viscosity and absorbance were measured to monitor the stability. The work of [74], in which the viscosity and electrical conductivity of DIW and EG-DIW (50:50) Al₂O₃-Fe₂O₃ (25:75) nanofluids (0.05–0.75 vol %) were measured at 20–50 °C, showed that the HNFs were prepared by ultrasonication for 2 h at frequency and amplitude of 70% while the dispersion fractions for DIW and EG-DIW-based HNFs were 1.1 (SDS) and 1.2 (SDBS), respectively. The preparation parameters and their values reported in these studies are noticed to be adequate to reproduce the results published by the authors.

The stability, thermo-optical properties (extinction coefficient and viscosity), and performance of DW-based CuO, Al₂O₃, and CuO-Al₂O₃ nanofluids as working fluids in a direct solar absorption collector were measured [80]. To improve the stability of the MNFs and HNFs, sodium hexa meta phosphate was used as a surfactant and UV-visual spectrophotometry and visual inspection were used to check their stability. To prepare the nanofluids, optimum pH, surfactant mass concentrations, and sonication durations of 8–9, 1.5, and 100–120 min; 7–8.2, 0.25–0.5, and 45 min; and 7.5–8.5, 1.25, and 100–120 min were reported for CuO, Al₂O₃, and CuO-Al₂O₃, respectively. Ultrasonication at 24 kHz and

amplitude of 70% was performed to reduce agglomeration and enhance stability. The details of the preparation parameters given by the authors are adequate to reproduce this work. In a similar study by Menbari and co-authors using the same NPs and HNPs but different base fluids of EG and EG-water (50:50), optimum pH (6.5–7.5 (Al₂O₃), 8.5–10 (CuO), and 7–8.2 (CuO-Al₂O₃)), surfactant mass concentration (0.25–0.5 (Al₂O₃), 1.65 (CuO), and 1.5 (CuO-Al₂O₃)), and sonication duration (55 min (Al₂O₃), 120 min (CuO), and 100–120 min (CuO-Al₂O₃)) were reported for EG-based MNFs and HNFs [170]. In addition, optimum pH (7–8 (Al₂O₃), 8–9 (CuO), and 7.2–8.5 (CuO-Al₂O₃)), surfactant mass concentration (0.25–0.5 (Al₂O₃), 1.75 (CuO), and 1.35 (CuO-Al₂O₃)), and sonication duration (40–50 min (Al₂O₃), 120 min (CuO), and 100–120 min (CuO-Al₂O₃)) were published for EG-waterbased MNFs and HNFs. With the preparation conducted at a frequency of 24 kHz and amplitude of 70% using an ultrasonicator, a detailed experimental procedure to replicate the experiment and findings is given by the authors.

Sharifpur and co-authors prepared TiO_2/DIW nanofluids (0.05–0.6 vol %) used to study the thermo-convection behavior of these thermal fluids in a rectangular enclosure using a one-step strategy [9]. The concentrated TiO_2/DIW nanofluid (15 wt %) was diluted to the required volume concentrations for the study and ultrasonicated for 3 min at 80% amplitude and 0.7 cycle time for homogenization. Using these parameters, this experiment can be repeated. Stability was improved by adjusting the pH to 9.5 and established using absorbency test, visual observation, and viscosity measurement. Recently, the fuel properties, performance, and emission characteristics of nano-fuels (30% biodiesel (50% palm and 50% sesame, 70% diesel, 10% dimethyl carbonate, 5% diethyl ether, NPs (100 ppm CNT and 100 ppm TiO₂ and surfactant (SDS)) on a diesel engine test rig were studied [171]. The nano-fuels were formulated by adding 20 mg of SDS to the fuel–NP mix and stirred for 30 min at 2000 rpm and sonicated for 30 min at 30% sonication amplitude with 3 s active and 2 s idle sonication mode. The details of the preparation parameters provided are found to be adequate to reproduce this study and achieve the same results.

To investigate the effect of Fe₂O₃ nanoparticles on the performance, combustion, and emission behavior of diesel plus Mahua methyl ester–diesel–pentanol, the nano-fuel was prepared by sonication using frequency of 45 kHz, duration of 30–45 min, revolution of 350 rpm, and temperature of 70 °C [172]. The provided preparation details are sufficient to reproduce the experiment. To examine the influence of different nano-sizes (8 and 20 nm), weight concentrations (0.01–0.2 g/L), and sonication time (30 and 120 min) on the AC breakdown voltages of CNT–transformer oil nanofluid, the samples were stirred for 30 min at 520 rpm and sonicated using amplitude of 10%, 5 s pulse on, 3 s pulse off, and 120 min to obtain stable test samples [133]. The stirring and sonication parameters provided are adequate to replicate this study.

4. Future Research Outlook and Conclusions

A survey of published works engaged in nanofluid research shows that ultrasonication, stirring, and use of surfactants are the principal nanofluid preparation characteristics. Figure 11 shows that generally, there are six approaches to nanofluid preparation, which are related to the two-step method. Figures 12 and 13 present the different values of thermal conductivity enhancement and relative viscosity of aqueous Al₂O₃ and other nanofluids as a function of nanoparticle volume fraction, respectively. The divergence in viscosity and thermal conductivity values for different types of nanofluids can be inferred from these figures (Figures 12 and 14). In addition, Figure 15 reveals the divergence in viscosity data for different nanofluids as the dependent of volume concentration and temperature. From Figures 12–14, the disparity in the results (thermal conductivity enhancement, relative viscosity, and viscosity) can be attributed primarily to the preparation characteristics (for a similar type of nanofluid), which include diverse sonication and stirring parameters and surfactant concentrations and the types of surfactants used in the experiment, if any. This result disparity is outside the effect of concentration, temperature, nano-size, and shape of NPs.



Figure 11. Nanofluid preparation approaches in the literature.



Figure 12. Schematic diagram for the dependence of the thermal conductivity of nanofluids on volume concentration in various studies [173].



Figure 13. A schematic diagram for the dependence of viscosity on the volume concentration of nanoparticles in various studies [173].



Figure 14. Divergence of viscosity data of different nanofluids under varying temperature and volumetric concentration [174].



Figure 15. Parameters for nanofluid preparation for different preparation approaches.

In Figure 15, the parameters involved in the ultrasonication, stirring, and surfactant deployment for nanofluid preparation are listed. These parameters have been reported in the literature to be associated with the preparation of nanofluids [84,86,87,96,126]. However, the need to optimize these parameters (those applicable) is important and has been established in the literature to be attained at optimum stable conditions for the HNF or MNF candidate. For ultrasonication, the amplitude, duration, frequency, and temperature of the sample, and the mode of sonication (continuous or pulse) must be optimized depending on the type of ultrasonicator deployed in the experiment. That is why it is also of the essence for authors to report the type (probe, centrifugal, and shaker) and specification of ultrasonicators used in nanofluid studies, as this will reveal the possible parameters to be optimized and thus enhance the reproducibility of the experiment, which would consequently assist in obtaining convergence of results. Speed, duration, and temperature of sample stirrer are parameters important to stability and ultrasonication, while surfactant concentration is key when a surfactant is used in the experiment to improve the stability of nanofluids. Optimizing these parameters is essential to achieving optimum stability conditions. The stirrer type (mechanical, magnetic, etc.) is crucial in addition to the specification, as both influence the stability of nanofluids. Taking the temperature of the sample during stirring and ultrasonication is beneficial, if possible, as it would further help in reproducing the experiment.

It can be deduced from the survey of the literature reviewed in this work and publications on nanofluid preparation and stability that there is still no standard procedure for the formulation of nanofluids based on the two-step strategy which involves the deployment of sonication. The diverse results published in the open literature and the various methods deployed by different authors to formulate nanofluids can be strongly linked to the different nanoparticles and base fluids with various chemical bonding structures and thus, with diverse stability mechanisms. The use of a suitable and more generalized formulation procedure is envisioned to promote more stable nanofluids and better results in terms of the different applications of nanofluids [133].

In view of the challenge of serious disparity in the result (rheological, optical, thermophysical, fuel, and anti-wear properties; supercooling, photothermal conversion, engine, lubrication, combustion, emission, and thermo-hydraulic performance)-especially for the same nanofluid—noticed in nanofluid studies and the inadequacy of preparation detail/procedure to reproduce experiments as established from the literature, possible and future convergence of results is anticipated to be an achievable feat only if there is a paradigm shift in the way nanofluid preparation is reported and performed. Towards attaining result convergence in nanofluid studies, authors are to provide detailed preparation parameters such as ultrasonication, stirring, and surfactant concentration parameters (see Figure 15 and Table 2) to facilitate the smooth reproduction of such experiments. The current trend of publications on nanofluid studies does not give room for experiment/result reproduction. However, experiments are only reproducible when detailed procedures are provided. It is expected that conformity in nanofluid preparation through the provision of detailed preparation information and optimization of the same would promote uniformity of results. For convergence, reliability, and accuracy of nanofluid/nano-fuel properties, the authors must give details of the measuring instruments. Figure 16 gives the names of measuring instruments for the different thermal properties of nanofluids.



Figure 16. Instruments for measuring different thermal properties of nanofluid.

Year	Nanofluid	Concentration	Application	Stirring	Sonication	Surfactant	Sonicator Name/Type	Reference	Stability
1998	Al_2O_3 and TiO_2 /water	1–10 vol %	Forced convection in tube	2 h @ 10,000 rpm	-	NU	-	Pak and Cho [168]	Visual
2019	Al ₂ O ₃ and WO ₃ /DW and paraffin	0.1–5% vol.	Nano-lubricant	30 min @ 500 rpm	1 h @ 60% amplitude with 0.5 s (pulse on)	NU	-	Dehghani et al. [169]	ZP
2021	MWCNT-Fe ₂ O ₃ (20:80)/DIW	0.1–1.5 vol %	Property	-	2 h @ 70% amplitude and 70% frequency	SDS (0.5 dispersion)	Hielscher/probe	Giwa et al. [81]	UV and viscosity
2016	Al ₂ O ₃ (20, 80, and 100 nm)/glycerol	1–5 vol %	Property	-	6 h (20–30 nm and 3 h (80 and 100 nm) @ 75% amplitude with 0.8 s (pulse active) and 2 s (pulse idle)	NU	Hielscher ultrasonic processor (UP200S, 200 W, 24 kHz)	Adio et al. [72]	ZP
2020	Al ₂ O ₃ -MWCNT (90:10–20:80)/DIW	0.1 vol %	Property	-	2 h @ 70% amplitude and 70% frequency	SDS and dispersion fraction of 1	Hielscher UP200S (400 W and 50 Hz)	Giwa et al. [75]	Visual, viscosity and UV
2020	Al ₂ O ₃ -Fe ₂ O ₃ (25:75)/DIW and EG-DIW (50:50)	0.05–0.75 vol %	Property	-	2 h @ 70% amplitude and frequency	SDS (DIW @ dispersion fraction of 1.1 and SDBS (EG-DIW @ dispersion fraction of 1.2	Hielscher UP200S (400 W and 50 Hz)	Giwa et al. [74]	Visual and UV
2016	CuO, Al ₂ O ₃ , and CuO-Al ₂ O ₃ /DW	-	Property/solar collector	-	100–120 min, 45 min, and 100–120 min for CuO, Al ₂ O ₃ , and CuO-Al ₂ O ₃ @ 70% amplitude and 24 kHz	1.5, 0.25–0.5, and 1.25 for CuO, Al ₂ O ₃ , and CuO-Al ₂ O ₃	Hielscher (UP-200S)	Menbari et al. [80]	UV and visual
2016	CuO, Al ₂ O ₃ , and CuO-Al ₂ O ₃ /EG and DW-EG (50:50)	-	Property/solar collector	-	55 min (Al ₂ O ₃), 120 min (CuO), and 100–120 min (CuO-Al ₂ O ₃) for EG and 40–50 min (Al ₂ O ₃), 120 min (CuO), and 100–120 min (CuO-Al ₂ O ₃) for EG-DW @ 70% amplitude and 24 kHz	0.25–0.5 (Al ₂ O ₃), 1.65 (CuO), and 1.5 (CuO-Al ₂ O ₃) for EG and 0.25–0.5 (Al ₂ O ₃), 1.75 (CuO), and 1.35 (CuO-Al ₂ O ₃) for EG-DW	Hielscher (UP-200S)	Menbari et al. [170]	UV and visual
2018	TiO ₂ /DIW	0.05–0.6 vol %	Natural convection	-	3 min @ 80% amplitude and 0.7 cycle time for one-step method	NU	Hielscher ultrasonic processor (UP200S)	Sharifpur et al. [9]	Visual, UV, and viscosity
2020	50% palm and 50% sesame, 70% diesel, 10% dimethyl carbonate, 5% diethyl ether, 100 ppm CNT and 100 ppm TiO ₂	-	Nano-fuel	30 min @ 2000 rpm	30 min @ 30% amplitude with 3 s (pulse on) and 2 s (pulse off)	SDS (20 mg)	Q500 sonicator (Qsonica, 20 kHz and 500 W)	Mujtaba et al. [171]	-

 Table 2. Future possible paradigm shift for nanofluid preparation.

As the stability of MNFs and HNFs is of utmost importance, their monitoring is crucial in nanofluid studies. Progression in nanofluid research has led to the hybridization of nanofluids, resulting in a new challenge in nanofluid stability. The advent of green nanofluids [175–180] and bio-based thermal fluids [181] for different applications and the mixture of the same [182,183] with currently available nanofluids also presents a fresh challenge to their stability which can affect their properties and application performance. In addition, there is a need to streamline nanofluid stability monitoring for conformity. The visual inspection method (non-scientific) in addition to ZP or UV–vis spectrophotometry and other techniques should be reported for nanofluid studies. Furthermore, stability must be ensured before and after the experiment to establish the degree of stability during the experiment.

The following conclusions are made based on this present study.

- 1. Preparation is key to nanofluid research, but stability is more important, especially where the nanofluid application is involved.
- 2. Owing to the variant in NPs and HNPs, base fluids, and concentration (weight or volume), achieving stability is an uphill task as stability conditions differ for each formulated MNF or HNF.
- Preparation of nanofluids involved cases of (i) sonication with and without surfactants, (ii) stirring and sonication with and without surfactants, and (iii) stirring with and without surfactants.
- 4. The propensity to reproduce nanofluid experiments and results is not reflected and entrenched in the current volume of published works and there is an urgent and pressing need to change the status quo by providing detailed experimental procedures concerning nanofluid preparation that can successfully lead to repetition of such reported studies.
- 5. There is a future need for convergence of results in nanofluid studies, which could be attained by the provision of detailed preparation parameters of stirring, surfactant concentration, and sonication involved in the studies and optimizing these parameters to achieve optimum stability conditions for better results.
- 6. Stability of nanofluids is to be measured before and after the experiments for further verification of the level of stability.

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