Thermal Conductivity of Al_2O_3 Nanofluid Utilizing Cross-linked Polyacrylic Acid (PAA) as the Base Fluid: An Experimental Study

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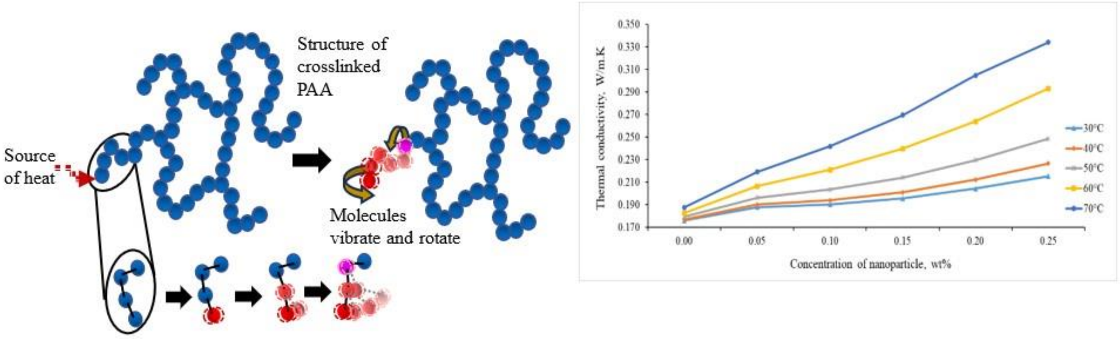
GRAPHICAL ABSTRACT

Synthesis of crosslink PAA solution

Heat transfer mechanism of PAA solution



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The thermal conductivity was measured for Al_2O_3 nanofluid using a newly developed polymeric base fluid. The novel base fluid of cross-linked polyacrylic acid (PAA) solutions was synthesized *via* radical polymerization using a distinct deep eutectic solvent (DES). Five weight concentrations of Al_2O_3 nanoparticles, 0.05, 0.10, 0.15, 0.20, and 0.25 wt%, were dispersed in the polymeric fluid *via* two dispersing techniques. In the first step, the nanoparticles were stirred using magnetic stirring for 1 h, followed by the sonication technique for another hour to ensure the nanoparticles were well suspended in the base fluid. A KD2 Pro thermal analyzer measured the thermal conductivity of each concentration for the temperature from 30 to 70 °C. The experimental data demonstrated a correlation between thermal conductivity and nanoparticle weight fraction. The results showed that the thermal conductivity increased with the increment of Al_2O_3 concentration for all set temperatures. The study revealed that the polymeric base fluid could replace the conventional base fluid since the thermal conductivity results were comparable with those reported in the literature.

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Keywords: Thermal conductivity; Base fluid; Polymeric fluid; Nanofluid; Cross-linked polyacrylic acid; Polymer solution; Al_2O_3 *nanoparticle*

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INTRODUCTION

The term “nanofluid” was first used in 1995 by Choi and Eastman (1995) to characterize a mixture of tiny particles suspended in ordinary liquids such as ethylene glycol, water, or oil. Since then, researchers have shown interest in the thermal and fluid sciences related to such fluids. In thermal engineering, nanofluids have been applied to solar energy, nuclear reactors, medicinal applications, vehicles, and electronic equipment (Saidur *et al.* 2011; Mahian *et al.* 2013; Surakasi *et al.* 2021). Switching to nanofluids from conventional heat transfer fluids is desirable since the conventional fluids have lower thermal conductivity than the nanofluids when incorporating solid nanoparticles (Ali *et al.* 2022; Venkataramana *et al.* 2022). Employing a fluid with a higher thermal conductivity accelerates the heat transfer pace and creates a smaller system or device. According to the literature review, one of the most often employed nanoparticles for creating nanofluids is aluminum oxide (Al_2O_3) (Gao *et al.* 2020; Joseph Arun Prasath *et al.* 2023).

Research on the thermal conductivity of nanofluids containing Al_2O_3 nanoparticles has been reviewed in the literature. Types of base fluids and concentration of Al_2O_3 significantly impact the thermal conductivity result. Research shows that increasing the concentration of Al_2O_3 nanoparticles in water can substantially enhance the thermal conductivity of the nanofluid (Chintala *et al.* 2020). A study by Kwek *et al.* (2010) shows that adding 1 to 5% volume fraction of Al_2O_3 nanoparticles in water can increase the effective thermal conductivity by 6% to 20% at room temperature. Xiang *et al.* (2019) also demonstrated an 18% increase in thermal conductivity with a 1% volume fraction of Al_2O_3 in mineral oil-based nanofluids. Similarly, Settino *et al.* (2022) showed that the use of synthetic oil-Al_2O_3 nanofluid led to a 7.6% increase in the thermal efficiency of the receiver, indicating a positive impact of nanofluids on thermal properties. Wanatasanappan *et al.* (2022) found that a palm oil-based nanofluid with a 0.6% mass concentration of Al_2O_3 hybrid nanoparticles exhibited a 27.5% enhancement in thermal conductivity.

The variation of nanofluid thermal conductivity has also been studied by Mostafizur *et al.* (2014). The study involved the production of nanofluids by dispersing Al_2O_3 nanoparticles in methanol as the base fluid, with nanoparticle volume fractions of 0.05%, 0.01%, 0.5%, 0.1%, and 0.15%. Their finding revealed that the thermal conductivity of the methanol-based nanofluids increased with higher particle volume fractions, and this thermal conductivity was compared to that of pure methanol. Across all concentrations, Al_2O_3/methanol nanofluids displayed higher thermal conductivity compared to pure methanol, with an overall increase of approximately 28%. In a separate study, Srinivasan et al. (2021) demonstrated that Al_2O_3/ethylene glycol nanofluids provide greater heat transfer enhancement compared to Al_2O_3/water nanofluids. Similarly, Kumar and Sahoo (2019) observed that the thermal conductivity of Al_2O_3 hybrid nanofluids varies between ethylene glycol and propylene glycol (PG) base fluids.

This indicates that the choice of base fluid is crucial, as it significantly impacts the thermal properties and performance of the nanofluid. Consequently, there is growing interest in exploring nanofluids by dispersing Al_2O_3 in non-conventional base fluids. To date, no studies have reported on the thermal conductivity of polymer solution-based nanofluids. Therefore, in this recent study, a crosslinked polyacrylic acid (PAA) solution was synthesized using deep eutectic solvent (DES) (Fauzi *et al.* 2023) as the solvent. DES was employed to reduce the freezing point of the nanofluid. As a result, the polymer solution-based nanofluid, in contrast to those with conventional base fluids, is able to remain in a liquid state for a wide range of temperatures, including those below 0 ℃, without undergoing crystal formation or freezing, thus preserving its physical properties. The cross-linked structure of the polymer, on the other hand, facilitates conductive heat transfer. Meanwhile, the bonding formed between nanoparticles and PAA molecules creates a heat-conductive pathway, enhancing the heat transfer performance of the nanofluid system. Apart from that, these bonds can prevent the non-uniform dispersion of the nano-filler in the polymer matrix. These provide significant advantages for the newly developed base fluid over conventional base fluids.

EXPERIMENTAL

Materials

N,N-Diethylethanolammonium chloride and ethylene glycol, supplied by Nacalai Tesque (Japan), were used to produce DES, with respective commercial-grade purities of

99.8% and 98%. Al_2O_3 nanoparticles, with a size of less than 50 nm, were supplied by Merck company (Germany). The other materials utilized in this experiment were sourced from Sigma Aldrich.

Base Fluid Preparation

Cross-linked PAA solution was produced through a radical polymerization method. The polymerization procedure was carried out in two steps. The first step was to form solution A, followed by the formation of solution B in the second step. To prepare solution A, purified acrylic acid monomer (AA) was dissolved in DES without heating. While in a separate beaker, the same quantity of DES was used to dissolve APS. The APS/DES solution was mixed into the AA/DES solution and stirred using a mechanical stirrer at 300 rpm for 30 min. This reaction was carried out on a heating plate at 65 °C.

MBA was added to solution A. Stirring was continued with the same mechanical stirrer speed and temperature. The resulting mixture was solution B, which is a cross-linked PAA solution. Since the desired amount of cross-linked structure was low, the amount of MBA added to solution A was varied. Apart from that, the amount of AA and the reaction time were also changed. The polymerization reaction was stopped *via* the quench technique by adding 1.0 mL of 2% hydroquinone solution into solution B at the end of the reaction time. Immediately after inhibition, solution B was immersed in an ice-water mixture for 10 min. This solution was kept cold before undergoing analysis.

Nanofluid Preparation

A special dispersion technique of nanoparticles was required to create a suspension with acceptable stability. This was necessary to avoid sedimentation and agglomeration of nanoparticles in the base fluid. Nanofluid was prepared by dispersing Al_2O_3 nanoparticles in the synthesized cross-linked PAA solution at concentrations of 0.05, 0.10, 0.15, 0.20, and 0.25 wt%. Nanoparticles were weighed to the respective concentrations and were gradually added to the PAA solution. The mixture was stirred using a magnetic stirrer for 1 h. Afterward, the suspension was sonicated for another hour using a probe sonicator to break down the agglomeration of nanoparticles. Utilizing these two dispersion steps resulted in more stable and uniform nanofluids. No sedimentation in the sample was observed with physical observation for more than 7 d.

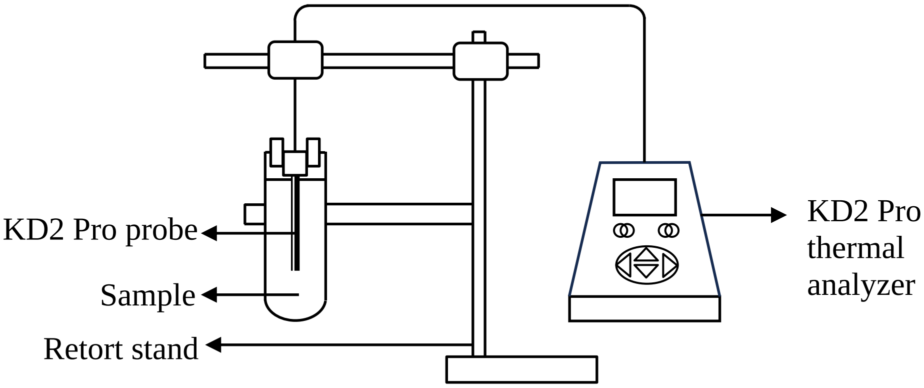

Fig. 1. Schematic diagram of the KD2 Pro thermal properties analyzer

Measurement of Thermal Conductivity

The thermal conductivity of PAA/Al_2O_3 nanofluids at various concentrations and temperatures ranging from 30 to 70 °C was measured using a KD2 Pro instrument, which operates based on the transient hot wire method. The KD2 Pro takes measurements every second over a 90-second cycle, with an accuracy of ± 5%. Each sample was tested three times, and the average value was recorded. A hot water bath was employed to maintain a stable temperature, and a thermometer with an accuracy of 0.1 °C was used for temperature measurement. Figure 1 is a schematic diagram of thermal conductivity measurement using a KD2 Pro thermal analyzer.

RESULTS AND DISCUSSION

The thermal conductivity test was conducted on the base fluid (cross-linked PAA solution) without nanoparticles. The results were compared with the thermal conductivity of the nanofluid at nanoparticle concentrations ranging from 0.05 to 0.25 wt% and temperatures varying from 30 to 70 °C. The thermal conductivity of the cross-linked PAA solution at 0 wt% Al_2O_3 nanoparticles was 0.176 W/m.K at the initial temperature of 30 °C. However, adding nanosolid materials (Al_2O_3 nanoparticles) with higher thermal conductivity (30 W/m.K) can enhance the thermal conductivity of the produced nanofluid (Selvarajoo *et al.* 2024). The PAA-Al_2O_3 nanofluids showed a faster thermal response compared to the PAA solution without nanoparticles, achieving a 7% increase in thermal conductivity with the addition of 0.05 wt% Al_2O_3. The addition of 0.10 wt% Al_2O_3 increased the thermal conductivity by 8%. Similarly, at Al_2O_3 concentrations of 0.15, 0.20, and 0.25 wt%, the thermal conductivity increased by 11%, 16%, and 22%, respectively, compared to the thermal conductivity of the PAA solution without Al_2O_3 at the test temperature of 30 °C. At 40 °C, the thermal conductivity of 0 wt% nanoparticle was 0.177 W/m.K. By dispersing 0.05, 0.10, 0.15, 0.20, and 0.25 wt% Al_2O_3 nanoparticles into the PAA solution, the thermal conductivity increased by 7%, 10%, 14%, 20%, and 28%, respectively. The study continued at 50 °C, where the initial thermal conductivity at 0 wt% nanoparticle was 0.180 W/m.K, and it increased by 9%, 13%, 19%, 28%, and 38% when Al_2O_3 nanoparticles were dispersed into the base fluid at the same nanoparticle concentrations. The increments in thermal conductivity at this temperature were higher compared to 30 and 40 °C.

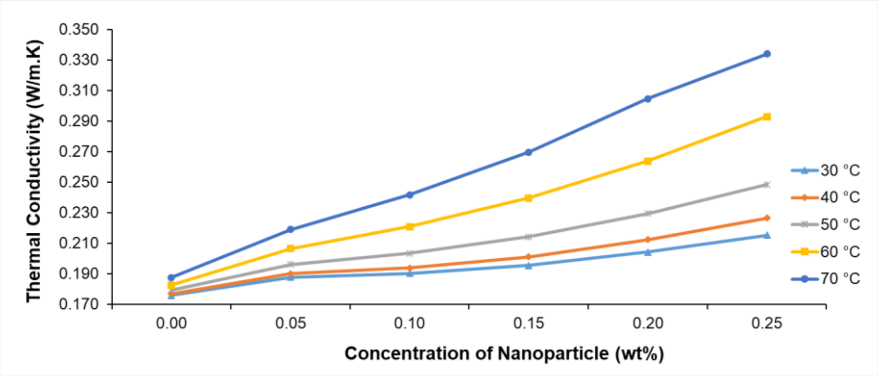

Fig. 2. Overall result for the thermal conductivity of PAA/Al_2O_3 nanofluids with respect to changes in Al_2O_3 nanoparticle concentration

To demonstrate the effect of Al_2O_3 nanoparticles on the thermal conductivity of nanofluids, the study continued at higher temperatures of 60 and 70 °C. The heat transfer rate of nanofluids was faster at higher temperatures, with the thermal conductivity of nanofluids increasing by 13%, 21%, 31%, 44%, and 60% at 60 °C, while 17%, 29%, 44%, 62%, and 78% at 70 °C, respectively, for the same nanoparticle concentrations. The data collected showed that the thermal conductivity of nanofluids increased with the presence of high thermal conductivity nanoparticles. The thermal conductivity further increased with increasing nanoparticle concentrations at all temperature conditions. Figure 2 shows the overall values of thermal conductivity enhancement for each nanofluid concentration.

Understanding the heat transfer mechanism in the polymer structure is important to provide insight into the heat transfer occurring in the nanofluid. Cross-linking is the bonds or sequences of short bonds that connect one polymer chain (PAA) to another through covalent bonds, making the PAA molecular structure rigid (Mo *et al.* 2024). Polyacrylic acid 'solution' refers to PAA with a low cross-linking degree, keeping the PAA in a solution state rather than a solid (Nardinocchi *et al.* 2024). The bonding between PAA chains always occurs irregularly, so the cross-linked PAA molecular structure is entirely amorphous and disordered (no dense lattice-like crystalline polymers). When a heat source is present, the heat will first reach the surface of the molecule closest to the heat source. This heat is then transferred to the adjacent molecule and then to the next molecule. In this condition, the heat does not propagate as a wave but slowly diffuses through the PAA network, causing irregular vibrations and rotations of all PAA molecules around their equilibrium positions and spreading to adjacent chains (Burger *et al.* 2016). These vibrations and rotations of the molecules will cause phonon scattering. Phonons are quantized lattice vibration waves that are the main carriers of thermal energy, contributing to heat capacity and conductive heat transfer in the condensed phase. They play an important role in the conversion of thermal energy (Yang *et al.* 2023). Figure 3 illustrates the amorphous structure of PAA and its heat transfer mechanism.

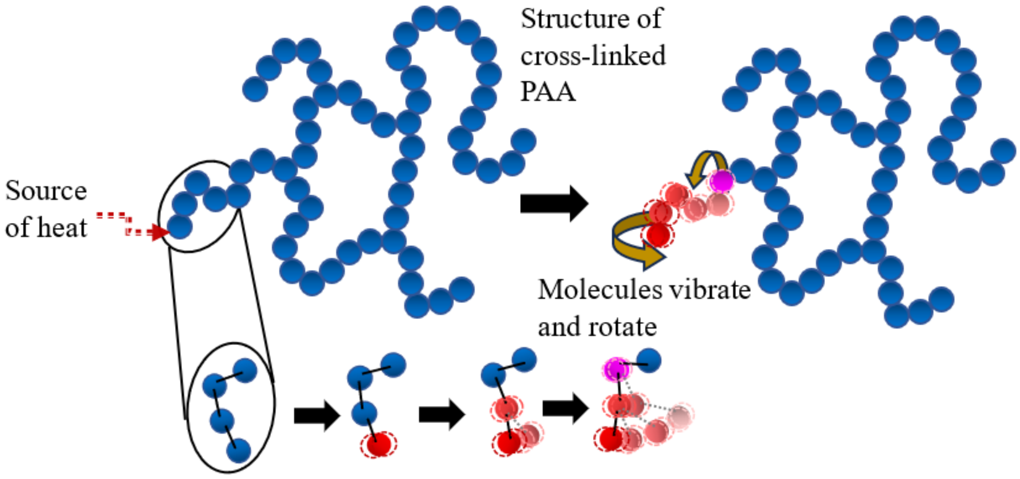

Fig. 3. Mechanism of thermal conductivity in amorphous polymers

A good thermal conductor has molecules arranged in an orderly manner, which can resemble crystalline polymers. However, the molecular arrangement of cross-linked PAA solution is disordered and amorphous. To understand the mechanism of good or poor thermal conductivity, Newton's pendulum is used as an example to understand the thermal conductivity of the PAA solution and compare it to the thermal conductivity of crystalline polymers (Chandrashekar *et al.* 2023). Figure 4 shows Newton's pendulum moved in an orderly manner, representing crystalline polymers (a), and a disordered movement of the pendulum representing cross-linked PAA solution (amorphous) (b). This figure illustrates the aspect of molecular vibrations in both structures. The ordered structure (a) will transfer the initial vibration to the opposite or adjacent surface quickly because the molecules are closely and orderly arranged. Conversely, in arrangement (b), the initial kinetic energy mostly scatters chaotically to other molecular structures due to the disordered arrangement of molecules, causing vibrations in each ball or molecule. Through the mechanism shown in Fig. 4, it can be summarized that the heat transfer technique involved in polymer molecules is conductive heat transfer.

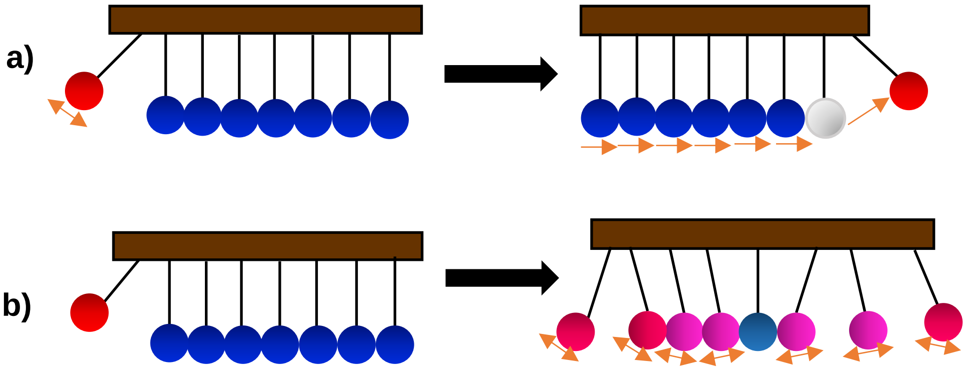

Fig. 4. Comparison of Newton's pendulum for the thermal conductivity of crystalline polymers (a) and amorphous polymers (b)

The morphology of the PAA structure suggests that it may not be effective for heat transfer through conduction. The amorphous aspect of the polymer, when viewed in the context of thermal conductivity, has weaknesses such as slow heat transfer. The disordered polymer chains prevent thermal energy from being quickly transferred across the polymer. This disorganized and random orientation contributes to phonon scattering, which causes thermal resistance and can lower the thermal conductivity of a material (He and Wang 2021). Therefore, the thermal conductivity of the cross-linked PAA solution at 0 wt% nanoparticles is low with the value 0.176 W/m.K.

Conversely, when nanoparticles are dispersed into the PAA solution, the thermal conductivity of the nanofluid increases. The increment of thermal conductivity due to the presence of nanoparticles results from the formation of conductive heat paths of Al_2O_3 nanoparticles throughout the PAA matrix, where these paths play a crucial role in phonon transfer (Chandrashekar *et al.* 2023). Phonon transfer occurs with non-covalent interactions such as ion bonding, hydrogen bonding, Van der Waals forces, or π-π interactions between the polymer matrix and the nano-filler (Kim and Choi 2021). These bonds can enhance the interface adhesion between the polymer matrix and the nano-filler (Jiao *et al.* 2023). Strong adhesion is essential to reduce interfacial resistance that can impede heat transfer in nanofluids. Moreover, these bonds can prevent non-uniform dispersion of the nano-filler, as this condition can lead to phonon scattering (Yang *et al.* 2021), which may result in reduced thermal conductivity, as illustrated in Fig. 5 (Awais *et al.* 2021). Therefore, the homogeneous and uniform dispersion of nanoparticles in the polymer matrix is another key factor in minimizing phonon scattering to achieve effective path flows of the nano-filler for heat transfer and facilitate efficient thermal energy transfer (Gordon *et al.* 2014).

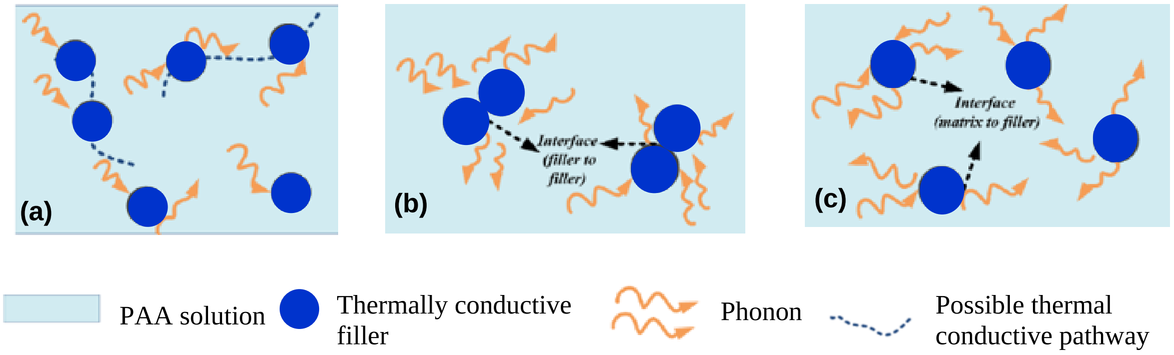

Fig. 5. Phonon scattering resulting from uneven nanoparticle arrangement.

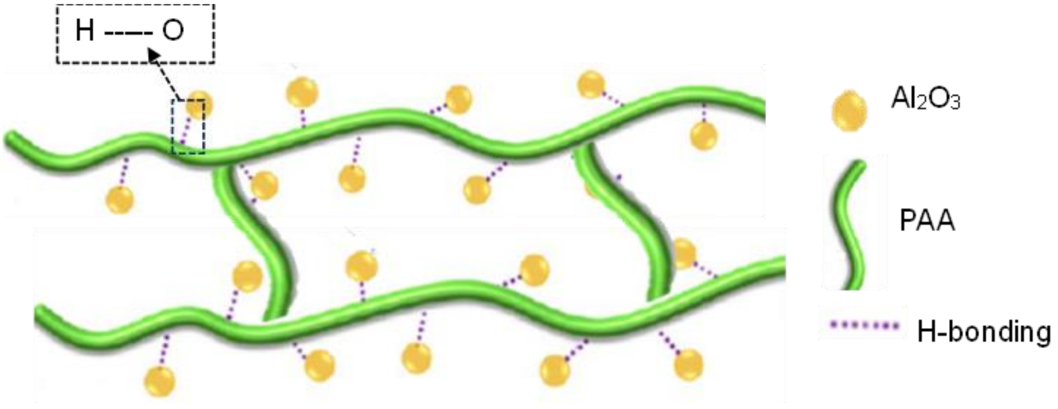

Fig. 6. Formation of hydrogen bonds between PAA and Al_2O_3 nanoparticles

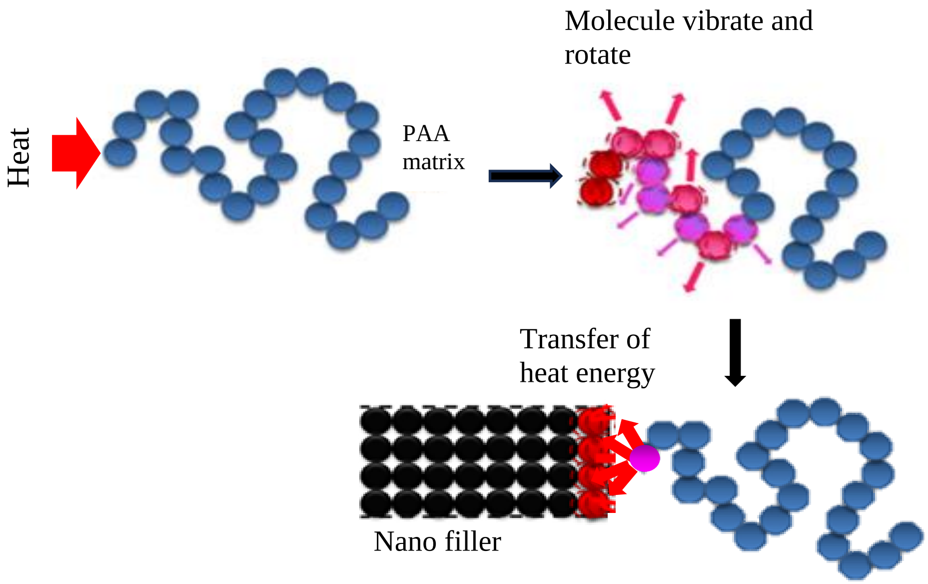

Fig. 7. Mechanism of thermal energy transfer from the PAA matrix to Al_2O_3 nanoparticles

The bond formation between PAA and Al_2O_3 is a hydrogen bond. Through this hydrogen bonding, the contact area between PAA and Al_2O_3 is large and minimizes heat resistance by reducing phonon scattering (Olmo *et al.* 2019). In this scenario, phonon transport is efficient, facilitating thermal energy transfer in the nanofluids. Therefore, as nanoparticle concentration increases, more hydrogen bonds form between PAA and Al_2O_3. This allows for better phonon transfer and faster heat transfer, resulting in increased thermal conductivity of the nanofluid. Figure 6 illustrates the formation of hydrogen bonds between Al_2O_3 and the PAA matrix, while Fig. 7 shows the heat transfer from the PAA matrix to Al_2O_3 nanoparticles.

CONCLUSIONS

1. It was found that the thermal conductivity of polymeric nanofluid increased between 7% to 78% with the increment of concentration of nanoparticles for each temperature that was considered.
2. The study suggests that the heat transfer mechanism in the newly developed polymeric nanofluid is primarily conductive.
3. Interaction between Al_2O_3 nanoparticles and poly(acrylic acid) (PAA) molecules enabled the formation of heat-conductive pathways, which could enhance heat transfer within the nanofluid and help prevent nanoparticle agglomeration.
4. Thermal conductivity values of newly developed nanofluid showed comparable results with the nanofluid generated from conventional base fluids. This suggests that the polymeric base fluid could replace the conventional base fluid

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