

The Effect of Surfactant on the Stability of Inorganic Salt Hydrated Phase Change Material Containing Graphene Nanoplatelet: An Experimental Investigation

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ABSTRACT

The use of nano phase change material (PCM) as a heat transfer agent has spread and expanded as its thermal conductivity is higher than the base PCM, so its use has been increased in PVT systems. In this study, inorganic salt hydrated PCM was used as a base material and Graphene nanoplatelet (GNP) as an additive to improve the thermal conductivity of the resulted nano-PCM and its stability was studied using Cetyl Trimethyl Ammonium Bromide (CTAB) surfactants. The main objective of the present study was to investigate the effect of surfactant on the stability of different GNP concentration in $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ PCM. The nano-PCM has been prepared using two-step method. The stability of nano-PCM depends on the surfactant used to slow the sedimentation of nanoparticles in the base PCM. The sedimentation process significantly reduces the thermal conductivity of nano-PCM and thus reduces heat transfer. The study results indicated that the employment of surfactant significantly affected the stability of nanoparticles added in the base PCM. The stability test ascertained the effectiveness of the surfactant applied in phase change material. With successive addition of surfactant, the stability of GNP in PCM was further enhanced to +49.3 mV of Zeta potential value.

1. Introduction

The stability of nanoparticle dispersions is an important factor in determining their ultimate consequence and interaction. The behaviour of sufficiently significant agglomerates of nanoparticles varies from that of well dispersed ones is well recognized [1]. Dispersion stability and interfacial

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properties are influenced by a variety of parameters, including: the intrinsic characteristics of the nanoparticles, and more particularly their surface properties [2]. The effect of strong Van der Waals interactions and cohesive forces between nanoparticles can cause nano-PCM to become unstable. Therefore, to break down these forces and develop stable nano-PCM, a variety of techniques and methods are vitally necessary. In order to prevent nanoparticle aggregation and increase the stability of nano-PCM, a variety of techniques have been employed, including functionalization, high-pressure homogenization, pH regulation, surfactant addition, ultrasonic agitation, magnetic stirring, and stirring [3]. The shape and concentration of nanoparticles, aggregation within the nano-PCM, and the length of sonication time utilized during preparation are the primary factors influencing the thermophysical properties of nano-PCM [4]. A crucial factor influencing the rheological and thermophysical behaviors of the nano-PCM is the stability of the nanoparticles suspended in the base material. Brownian motion leads particles to collide, resulting in cluster formation in the underlying material. Various internal forces between the base material and the nanoparticles control these cluster formations or aggregations, such as the Van der Waals forces of attraction between the particles [5]. The gravimetric and structural studies by Chinnasamy *et al.*, [6] confirm the stability of dispersed phase and base fluid in the presence of surfactant. The studies concluded that the PCM emulsion with mixed surfactant (Sodium dodecyl sulfate and Brij30) can be a potential heat transfer medium with improved heat storage and transportation capacity. Sheikh *et al.*, [7] enhanced the thermal stability of nano-PCM composite by employed the sodium dodecyl sulfate (SDS), sodium dodecyl benzenesulfonate (SDBS), and sodium stearyl lactylate (SSL) as the surfactants. In addition, the surfactants also used to measure the thermal characteristics in terms of indices such as thermal conductivity, thermal capacity, and time of reaching the reference temperature. Rehman *et al.*, [8] investigated the influence of eight distinct surfactants on the stability, rheological characteristics, and thermophysical properties of hybrid nanofluids (NFs) containing Aluminum oxide (Al_2O_3) and Titanium dioxide (TiO_2) in a Water-Ethylene Glycol (EG) mixture (60:40). The best surfactant for long run applications in terms of stability, viscosity and thermal conductivity are the objectives of the studies. As the result, polyvinylpyrrolidone (PVP) exhibited the highest stability among all surfactants, surpassing others in zeta potential and visual stability over 60 days. Research by Alawi *et al.*, [9] described that the thermal conductivity of pentaethylene glycol-treated graphene nano-platelets (PEG-GnP) and pentaethylene glycol thermally treated graphene (PEG-TGr) which were dispersed in distilled water nanofluids managed to achieve a respective enhancement of 32% and 31% at 50°C and 0.1% mass concentration. Meanwhile, a higher thermal conductivity than that of pure stearic acid was reported by Li *et al.*, [10] through the addition of different carbon additives mixed with graphene. Overall, it was summarised from the experiment that the addition of carbon additives (graphite-based nano-composite) led to the increase of thermal conductivity of the nano-composite by 12 times higher than that of pure stearic acid. Moreover, this is in agreement with the study conducted by Yavari *et al.*, [11] which conducted an experiment on uniform dispersion of graphene flakes nanoparticles. The result showed that the thermal conductivity of graphene composite increased by about 140% than that of pure 1-octadecanol without incurring a large reduction in phase change enthalpy. A possible explanation to this may be the expedition of the heat transfer and effectiveness of phonon that travels through the graphene fillers and between the flakes. On a similar note, Mehrli *et al.*, [12] reported that the thermal conductivity of a new form-stable composite phase change materials (PCMs) prepared through vacuum impregnation of paraffin within graphene oxide (GO) sheets were significantly enhanced from 0.305 to 0.985 (W/m.k). In addition, Du *et al.*, [13] the paper further investigated the effect of surfactants on nanofluid boiling by employing, cetyltrimethylammonium bromide (CTAB), sodium dodecyl sulfate (SDS), and polysorbate 20 (Tween 20). The heat transfer coefficients of the nanofluids with CTAB and with SDS were significantly

increased by 55.45% and 9.73% compared to the nanofluid without surfactant. According to Boldoo *et al.*, [14] the addition of CTAB surfactant positively influenced the stability of the slurry dispersion, ensuring a more even distribution of me-PCMs within the base fluid. The findings highlight the potential of me-SAC and me-SAL slurries as effective materials for TES applications. Significantly, me-SAL slurries outperformed me-SAC slurries in TES performance due to their greater latent heat energies during melting and solidification. A cationic surfactant called cetyltrimethylammonium bromide (CTAB) is used to increase nanofluid stability. It was discovered that the mean particle sizes of suspended nanoparticles in carrier fluid were 80 nm and 536 nm for nanofluid with and without surfactant respectively as reported by Mehta *et al.*, [15]. Li *et al.*, [16] employed various types of surfactants in $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ with gamma aluminium oxide ($\gamma\text{-Al}_2\text{O}_3$) nanoparticles in ensuring the stability of the particles in PCM. According to the study, CTAB with 1.0 wt.% had been proven as the best candidate among SDBS, SDS, and Span-80 surfactant. Regarding this matter, uniform and stable dispersion of nanoparticles in the PCM base solution had also been discovered as important driving factors in the enhancement of the thermal properties of the nano-PCM. However, a large surface area made it more difficult for nanoparticles to be uniformly dispersed in PCM solution. According to Ma *et al.*, [17], an overwhelming problem in uniform dispersion may occur due to the existence of even a few nanometers thick interfacial region. In the case of CNT reported by Choi *et al.*, [18], a small diameter in nanometer scale with a high aspect ratio (>1000) as well as a large surface area will contribute to a large interface area between the CNT and matrix, thus complicating the uniform dispersion. Surprisingly, there is no study regarding the CTAB surfactant effect on inorganic $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ /GNP nanocomposite PCMs reported.

In the present study, the GNP nanoparticles were embedded into the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ as base PCM to develop advanced $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ /GNP nanocomposite PCM. The study was further conducted to investigate the effect of surfactant on the stability of different GNP concentration in $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ PCM nanocomposite.

2. Two-Step Method

Two-step method is the most economic method to produce nanofluids in large scale, because nano-powder synthesis techniques have already been scaled up to industrial production levels [19]. According to N'Tsoukpoe *et al.*, [20], the two-step method is one of the more practical and effective ways of preparing the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ nano-PCM. In addition, the most extensively used approach for preparing nano-PCM is the two-step approach. Specifically, it should be noted that the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ material was produced through the dissolvent and crystallisation of industrial-grade CaCl_2 . Meanwhile, the dry powder nanoparticles employed in this process are first created using chemical or physical processes. The purpose of this activity is to synthesis the advanced nano-PCM in order to enhance thermal conductivity of PCM.

In addition, an ideal condition and solution of the advanced nano-PCM was the outcome obtained from this activity. The initial preparation of the homogeneously advanced nano-PCM involved the use of a magnetic stirrer at a constant temperature, 50°C in 10 minutes. Next, the graphene nanoparticle was added to the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ -based PCM to allow it to be mixed into the solution and continuously stirred for an extra 20 minutes. The process these steps is shown in Figure 1.

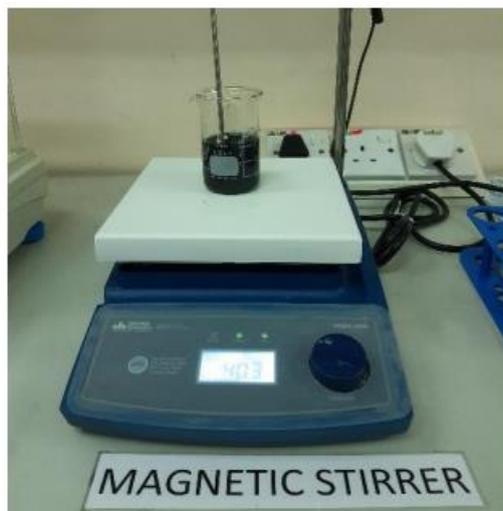


Fig. 1. Mixed solution of the advanced nano-PCM by using magnetic stirrer

Finally, ultrasonic vibration was applied in the preparation process for another 30 minutes as a control parameter to improve the dispersion stability of the mixture as well as minimise nanoparticle aggregation. Meanwhile, it is worth to note the importance of liquidity preservation which acts as a factor that can constant the vibrator temperature above the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ melting point during the preparation process. The ultrasonication apparatus for the advanced nano-PCM samples preparation is shown in Figure 2.



Fig. 2. Ultrasonication apparatus for samples preparation

The current research managed to record that the volume fractions of the nanoparticle were 0.1, 0.5, and 1.0 wt.% of GNP. Next, both the nucleating agent and surfactant were added to all of the advanced nanocomposite PCM and further sonicated for 60 minutes. The preparation method adopted in the present study is schematised in Figure 3.

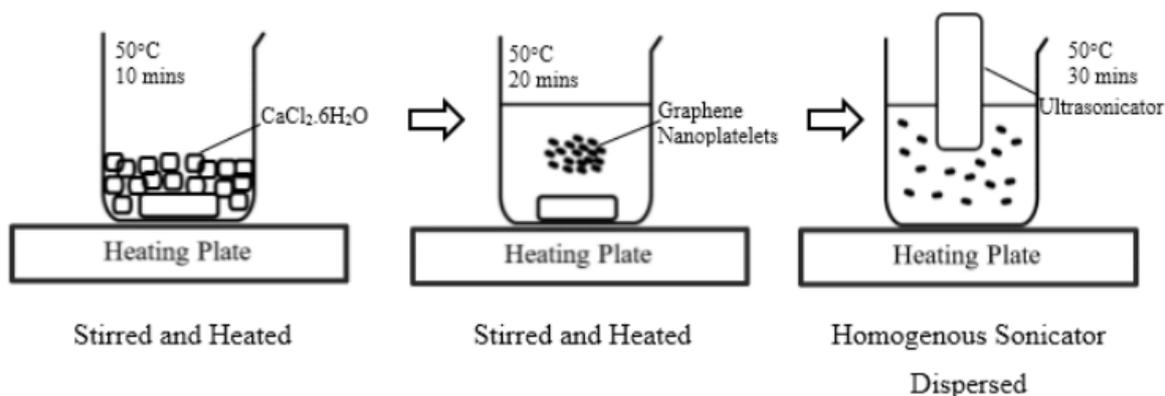


Fig. 3. Advanced nano-PCM, $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ -GNP sample preparation [21]

3. GNP Stability

The aim and purpose of exploring the effect of surfactant on GNP in the present study was to obtain the significant enhancement value of the advanced nano-PCM chemical dispersion stability. As illustrated in Figure 4, a visual sedimentation method was initially employed to observe the formation of nanoparticle agglomeration and sedimentation of GNP in PCM. Nevertheless, this method is considered unclear even though it can contribute a visible change versus time because it is directly the consequences of visual observation. In addition, it is caused by the absence of quantifying results that evaluate the degree of stability. Similarly, the dispersion stability of nanocomposite phase change material was verified using photo-image analysis reported by Al-Mahmodi *et al.*, [22].

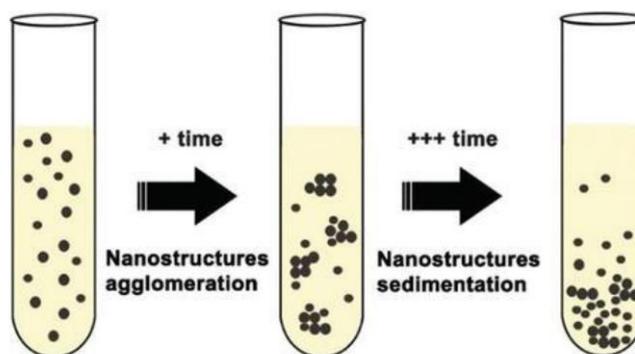


Fig. 4. Schematic of nanoparticles agglomeration and sedimentation over time [23]

As a result, the stability of nano-PCM had to be further determined in the present study using Zeta Potential particle analyser (Anton Paar) as shown in Figure 5. This approach was also implemented in the study by Pochapski *et al.*, [24] which utilized the Zeta potential and colloidal stability predictions for inorganic nanoparticle dispersions. Next, the stability of nano-PCM without the surfactant was also determined for comparison and benchmarking purposes. Finally, the enhancement of stabilised advanced nano-PCM was quantitatively evaluated. A spread and dislodge GNP in the PCM was a goal of this particular experiment. Li *et al.*, [16] employed CTAB as surfactant in $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ with gamma aluminium oxide ($\gamma\text{-Al}_2\text{O}_3$) nanoparticles in ensuring the stability of the particles in PCM. According to the study, CTAB with 1.0 wt.% had been proven resulted in uniform and stable dispersion of nanoparticles in the PCM base solution which had also been discovered as

important driving factors in the enhancement of the thermal properties of the nano-PCM. Table 1 shows the technical specification for the Zeta Potential particle analyser.

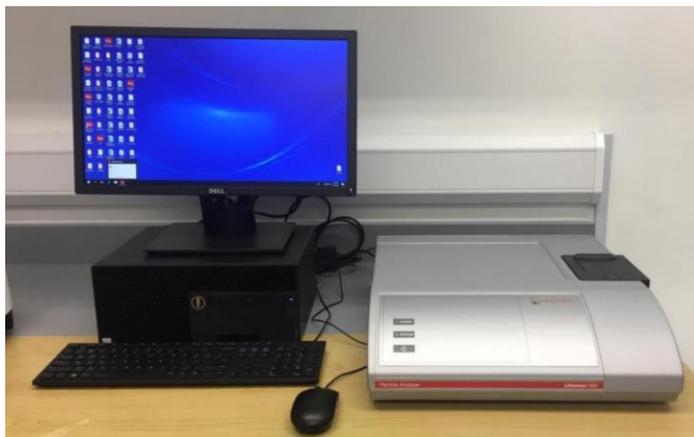


Fig. 5. Zeta Potential particle analyser (Anton Paar)

Table 1

Anton Paar Zeta Potential particle analyser specifications

Item	Specification
Measuring principle	Electrophoretic Light Scattering (ELS) / cmPALS
Measuring range	$\geq \pm 1000$ mV
Size range	3.8 nm to 100 μ m (diameter)
Accuracy	± 10 %
Repeatability	± 3 %

4. Result and Discussion

The heterogeneous properties of GNP in PCM are significant when the nanoparticle is in an unstable state. Figure 6 illustrates the poor dispersibility of GNP in salt hydrate PCM overtime for three different GNP concentrations (0.1, 0.5, and 1.0 wt.%). Hence, a non-covalent functionalisation approach was adopted in the current research for the purpose of enhancing the dissolvability of GNP without demolishing its inherent properties. Meanwhile, CTAB surfactant was utilised to increase the stability of GNP nanoparticle in PCM by reducing material adhesion as well as providing steric repulsions with the aim of counteracting the van der Waals attraction force. As can be seen from the figure, the nano-PCM with the employment of CTAB reports a more significant and stable diffusion after 14 days observation of the sedimentation method.

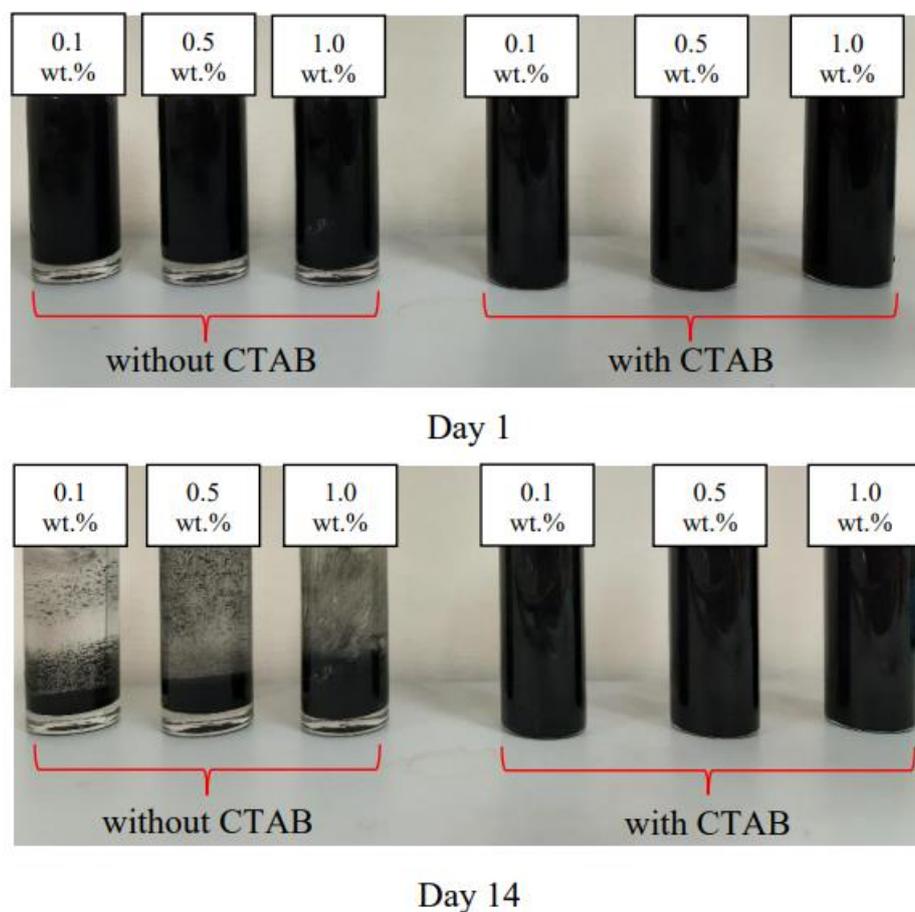


Fig. 6. Sedimentation method observation of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ -GNP with CTAB and without CTAB

In addition, Table 2 summarized the physically observation results for agglomeration and sedimentation analysis. The result shows that all concentrations of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ -GNP nanocomposites well dispersed and stable with the presence of CTAB surfactant.

Table 2
 Physically observed results for $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ -GNP stability analysis

Samples	GNP concentration, wt.%		
	0.1	0.5	1.0
Without CTAB	Agglomerated and sediment	Agglomerated and sediment	Agglomerated and sediment
With CTAB	No agglomeration and no sedimentation	No agglomeration and no sedimentation	No agglomeration and no sedimentation

Next, Figure 7 shows a quantitative enhancement of GNP stability in the PCM. The advanced nano-PCM without CTAB is recorded in Table 3 as 13 mV which indicates poor stability, while the advanced nano-PCM with the employment of CTAB significantly shows good stability with the recorded result of 49 mV. However, it is worth to note that the measurement was limited to 0.1 wt.% of GNP due to the equipment limitation.

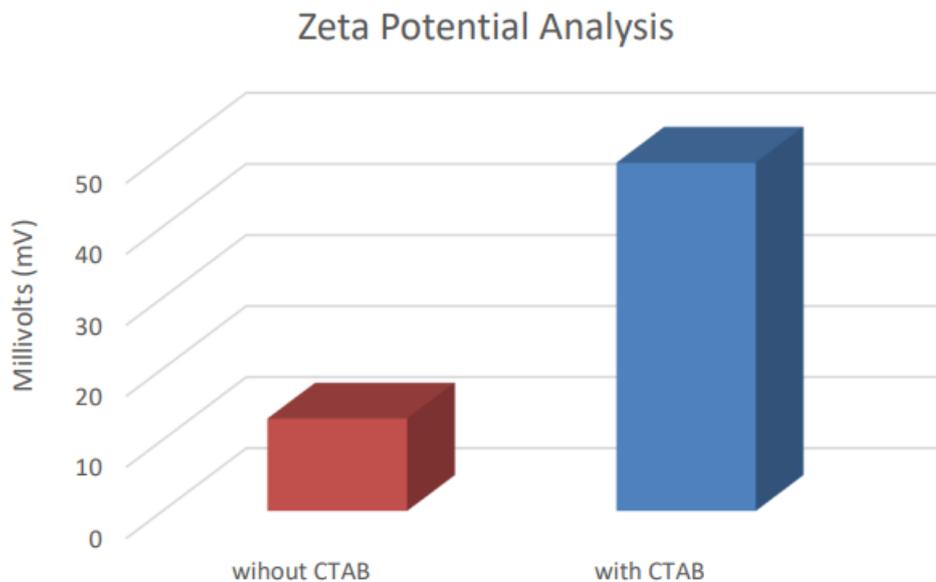


Fig. 7. Zeta potential of advanced nano-PCM with and without using surfactant (CTAB)

Table 3

Zeta potential values for both with and without CTAB embedded in nano-PCM	
Nano-PCM without CTAB	Nano-PCM with CTAB
13 mV	49 mV

According to Xian *et al.*, [25], higher concentration (0.5 and 1.0 wt.%) of graphene-based suspensions can lead to significant noises in the spectrum, followed by its naturally black appearance that tends to absorb the most transmitted light from a source. Apart from that, 0.1 wt.% of graphene-based nano-PCM was found to provide clearer results whereby this concentration managed to demonstrate all stability evaluations in the present study. In addition, Liu *et al.*, [26] reported that there was only a slight decrease in the zeta potential even though the concentration of graphene changed several times.

5. Conclusion

Agglomeration and sedimentation of GNP in the PCM was minimised as much as possible considering the importance of GNP stability in the current research. A significant finding to emerge from the present study refers to the requisition of surfactant (CTAB) as a stabilizer in the advanced nano-PCM. The present study demonstrated that GNP stability has been successfully proven through the implementation of surfactant in the advanced nano-PCM. The zeta potential result illustrated an increase in the stability of the GNP above the 30 mV value.

Moreover, further research regarding the role of surfactant would be worthwhile and interesting. The issue of segregation and agglomeration is an intriguing one which could be usefully explored in further research. There is, therefore, a definite need for the thermal stability of the NEPCM. Furthermore, the implications of invented synthesis method will be a route to the properties study of specific thermal characterization techniques which should be considered for further studies.

The current research has thrown up many questions in need of further investigation in enhancing the fundamental understanding of advanced nano-PCM for thermal energy storage application. A

recommendation to analyse the thermal properties with different concentrations (wt.%) of surfactant is a way forward the further study the effect of surfactant in advance nano-PCM.

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