Effect of Nano-Additives on Fracture Toughness of Engineering Materials: An Experimental Study / Original study

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تأثير الإضافات النانوية على متانة الكسر للمواد الهندسية: دراسة عملية

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Abstract

Structural integrity and service life of engineering components is based on fracture resistance. The paper presents an experimental study on the nano-additive that can be added to the locally-produced Iraqi engineering materials in order to enhance their fracture toughness. The research was inspired by the increasing demand in high-performance materials in the main sectors of the country such as infrastructure and energy besides the need to improve the properties of local resources. The tested material systems were two Portland cement composites and epoxy-based polymers. Nano-silica ($n-SiO_2$ nano-alumina ($n-Al_2O_3$) and multi-walled carbon nanotubes (MWCNTs) were added to these matrices in different weight ratios (0.5% to 2.0%). Parameters of the fracture toughness including planestrain fracture toughness (K_Ic) and the J-integral (J_Ic) were established according to the ASTM E399 and ASTM E1820 guidelines. The experimental evidence proves that fracture toughness is improved significantly in the presence of nanoparticles. In the case of cement composites, addition of $n-SiO_2$ in 1.5 wt.% resulted in the increase of K ic by up to 45%. MWCNTs loading of 0.5 wt.% increased J Ic by more than 70 in epoxy systems. Microstructural examination also indicated that the observed improvements were as a result of microstructural toughening processes, which include deflection of cracks, nanoparticles pull-out and bridging of microcracks. Such conclusions support the practical importance of nanotechnology in the improvement of the performance and reliability of engineering materials in Iraq, and provide a possible avenue towards lessening dependence on imported high-performance materials and improving the lifespan of national infrastructure.

Keywords: Fracture Toughness, Nanocomposites, Cement, Epoxy, Iraqi Materials, Experimental Testing.

المستخلص

تعتمد السلامة الهيكلية والعمر التشغيلي للمكونات الهندسية على مقاومة الكسر. تقدم هذه الورقة دراسة تجريبية حول الإضافات النانوية التي يمكن دمجها مع المواد الهندسية المنتجة محليًا في العراق بهدف تحسين متانة الكسر لديها. جاءت هذه الدراسة استجابةً للطلب المتزايد على المواد عالية الأداء في القطاعات الرئيسة في البلاد مثل قطاع البنية التحتية وقطاع الطاقة، إضافةً إلى الحاجة إلى تطوير خصائص الموارد المحلية.

شملت أنظمة المواد المختبرة مركّبين من إسمنت بورتلاندي وبوليمرات قائمة على الإيبوكسي. وتمت إضافة السيليكا النانوية ($n\text{-}SiO_2$) والألومينا النانوية ($n\text{-}Al_2O_3$) وأنابيب الكربون النانوية متعددة الجدران (MWCNTs) إلى هذه المصفوفات بنسب وزنية مختلفة تراوحت بين (0.5% – 2.0%). وقد تم تحديد معاملات متانة الكسر، بما في ذلك متانة الكسر في حالة الإجهاد المستوي (K_Ic) وتكامل J الحرج (J_Ic)، وفقًا لإرشادات المعيارين ASTM E399 وASTM E1820.

أظهرت النتائج التجريبية تحسنًا ملحوظًا في متانة الكسر عند وجود الجسيمات النانوية. ففي حالة المركبات الإسمنتية، أدى إضافة السيليكا النانوية بنسبة 1.5% وزنيًا إلى زيادة قيمة K_Ic بنسبة وصلت إلى 45%. كما أن تحميل أنابيب الكربون النانوية متعددة الجدران بنسبة 0.5% وزنيًا أدى إلى زيادة قيمة J_Ic بأكثر من 70% في أنظمة الإيبوكسي. وأوضحت الفحوصات المجهرية أن هذه التحسينات تعود إلى آليات تقوية بنيوية دقيقة، شملت انحراف الشقوق، وسحب الجسيمات النانوية، وتجسير الشقوق الدقيقة.

تدعم هذه النتائج الأهمية التطبيقية لتقنية النانو في تحسين أداء وموثوقية المواد الهندسية في العراق، كما توفر مسارًا واعدًا لتقليل الاعتماد على المواد عالية الأداء المستوردة وإطالة العمر الخدمي للبنية التحتية الوطنية.

الكلمات المفتاحية: متانة الكسر، المواد المركبة النانوية، الإسمنت، الإيبوكسي، المواد العراقية، الاختبارات التجريبية.

1. Introduction

Fracture mechanics is a quantitative description of the nature of failure of materials which include cracks or other flaws. The idea of fracture mechanics has its roots in the earlier work of Griffith in 1920 [1]. Fracture mechanics is the alternative to the conventional use of the strength-of-materials methods, which presuppose that the integrity of solids is fla.wless, since in the real world, practically every engineering construction possesses intrinsic defects [2]. These flaws are stress concentrators and when subjected to load can spread a disaster at stress levels that is many times less than nominal yield strength of the material. It is this phenomenon that has led to many engineering failures and the reason as to why designing to be fracture tolerant is extremely imperative [3]. Fracture toughness is a property used to measure the ability of the material to resist the growth of a crack, becoming a crucial element of contemporary structural integrity evaluation and failure analysis [4], [5].

The development of nanotechnology in the last few decades has led to a breakthrough in the field of materials science, and it has presented unprecedented possibilities to improve the mechanical characteristics of the traditional engineering materials [6]. Through adding particles that have a size in the nanometer range (nanoparticles) into a bulk matrix, one can form nanocomposites that have properties that are much better than that of the individual components [7]. Nano-silica ($n-SiO_2$),), nano- alumina ($n-Al_2O_3$), and carbon nanotubes (CNTs) are nanoparticles that are extremely strong, stiff and have a high surface area volumetric ratio. Being properly dispersed in a matrix such as cement or polymer, they can initiate special energy-dissipating processes at the nanoscales, therefore, slowing crack propagation and enhancing fracture resistance substantially [8], [9] see Figure 1.

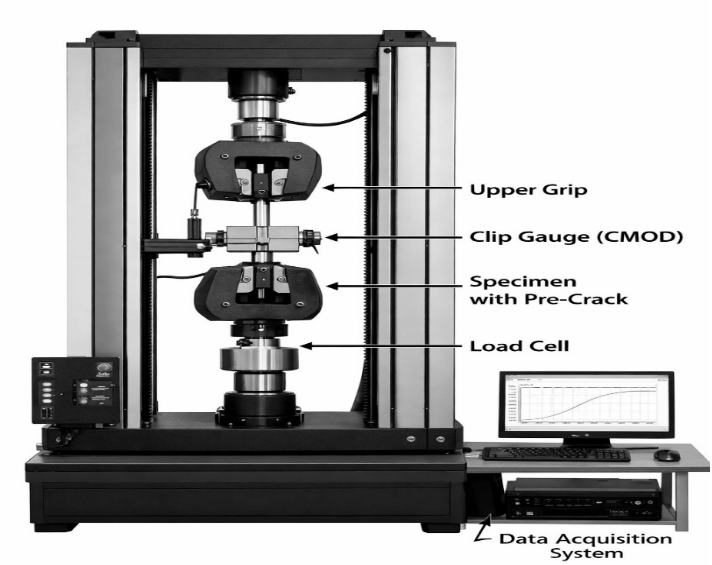


Figure 1. A typical universal testing machine configured for mechanical property evaluation, including fracture toughness tests [10].

The problem that has been identified and investigated in the current paper is the necessity to enhance the performance and durability of engineering materials sources and that used in Iraq. Oil and gas, construction and transportation are the main industries that are dependent on materials such as concrete and polymers. Nevertheless, conventional local materials are usually not high fracture resistance materials needed in the rigorous applications, which results in early failures, expensive maintenance, and use of expensive imported specialty products [11]. As an example, the stability of the fracture properties of oil well cement, which is very important in stability of drilling activities in Iraqi oil fields, is directly associated with the well cement integrity [12]. On the same note, concrete infrastructure that includes buildings and bridges should have the ability to resist cracking due to service load and environmental stress factors, which determines how long the structures will last [13]. Guidelines exist in materials testing through Iraqi Central

Organization of Standardization and Quality Control (COSQC) [14], although there is a huge gap with regards to the creation and characterization of highly advanced composites manufactured in Iraq.

This study is significant because it can offer a viable and cost-efficient measure towards improving the structural integrity of materials pertinent in the economic growth of Iraq. Through nanotechnology, some of the local sources of raw materials like the Babylon or Kufa cement factory [15], [16] can perhaps be enhanced into a high-performance material. This is in line with the national strategy to enhance the local industries, enhance the resilience of infrastructure, and develop local proficiency in materials science at the state of the art which has taken on increasing interest in Iraqi institutions such as the University of Technology and the University of Babylon [17], [18].

The study, consequently, works on the main hypothesis that on incorporating the selected nano-additives ($n-SiO_2$, Al_2O_3 and MWCNTs) under controlled conditions in Portland cement and epoxy resin sourced in Iraq, a measurable and significant increase in the fracture toughness of these materials will be observed. It is postulated that the enhancement will be reliant on the kind and amount of the nano-additive with an ideal loading prevailing per material framework. The study will try to test this hypothesis experimentally, in order to determine the underlying mechanism of toughening, thus, offering a scientific foundation to the practical implementation of the nanotechnology in the Iraqi engineering industry, see Figure 2.

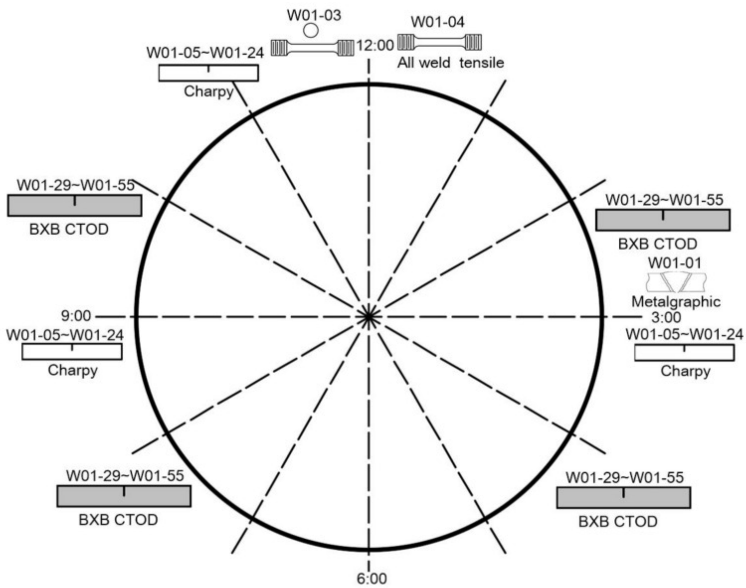


Figure 2. Schematic showing specimen extraction from a pipeline girth weld, a critical component in Iraq's oil and gas infrastructure where fracture toughness is paramount [19].

2. Theoretical Background and Literature Review

The concept of fracture mechanics gives the theoretical foundation of the relationship between the stress condition at a crack tip and the condition necessary to cause the crack to propagate. It is a wide-ranging field, with two subdivisions: Linear Elastic Fracture Mechanics(LEFM), and Elastic-Plastic Fracture Mechanics(EPFM), which relies on whether the crack tip has undergone plastic deformation or not.

2.1. Fundamentals of Fracture Mechanics

LEFM can be applied to brittle materials whereby plastic deformation is strongly concentrated in a limited area as compared to the crack size and specimen dimension [20]. The stress surrounding the end of a sharp crack may be defined in the form of one parameter the stress intensity factor (K), which summarizes the impact of applied load, the size, and geometry of the crack [20]. The overall shape of the equations of stress field in the vicinity of the point of crack tip is as follows:

$$\sigma_{ij} = (K / \sqrt{2\pi r}) * f_{ij}(\theta)$$

in which r is the distance to the tip of the crack, the angle is theta relative to the crack plane and fij(θ)is a dimensionless function of the angle [21]. Depending on the mode of loading, the stress intensity factor K varies. Three basic modes of crack movement are Mode I (opening mode), Mode II (in-plane shear) and Mode III (out-of-plane shear) [22]. Mode I is the most prevalent and most crucial in engineering practice. K can be written in that case:

$$K_I = Y\sigma\sqrt{\pi a}$$

where, - is the remote stress applied, a is the length of the crack and Y is a geometry factor that is non dimensional that takes into account the peculiarity of the body being cracked [21]. When KI attains a critical level, which is referred to as plane-strain fracture toughness, KIc, fracture is anticipated as shown in Figure 3. This value is regarded as a material property, which is the intrinsic resistance to brittle fracture under certain size and constraint requirements as specified in such standards as ASTM E399 [22] .

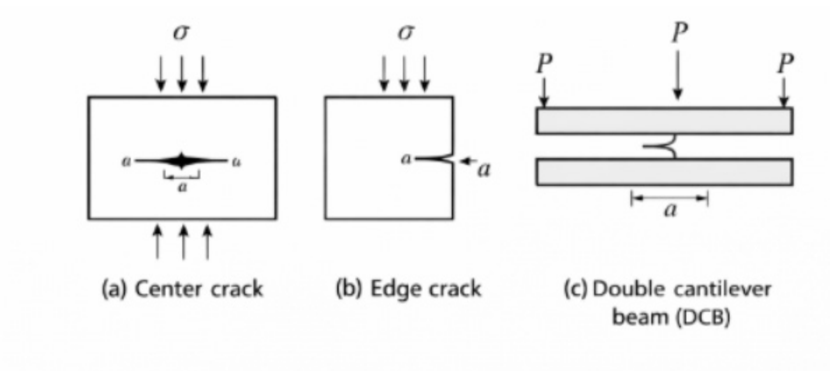


Figure 3. Common crack geometries used in fracture mechanics analysis: (a) center crack, (b) edge crack, and (c) double cantilever beam (DCB) [21].

In more ductile materials, in which large plastic deformation occurs before fracture, the assumptions of LEFM are broken. EPFM offers other parameters to define fracture toughness including the J-integral and the Crack Tip Opening Displacement (CTOD) [23]. J-integral is a measure of stress-strain field intensity in front of the crack that is associated with the energy release rate of a nonlinear elastic material. A critical value, JIc, is the beginning of steady growth of the crack. The standard ASTM E1820 is the composite standard of measurement of KIc , JIc and CTOD based on a single test [24].

2.2. Literature Review on Nano-Enhanced Materials

Nano-additives and their application in enhancing fracture toughness of polymers and cementitious composites has been widely discussed across the globe. In the case of epoxy resins, which are brittle in nature, nanoparticles have been demonstrated to bring in several toughening mechanisms. Rafiee, *et al.*, [25] saw a 65 percent increase in toughness of fracture in epoxy when 0.125 wt. percent of functionalized nanosheets of graphene is added to it. Domun, *et al.*, [26] studied the hybrid systems and realized that a mixture of 1D (f-MWCNTs) and 2D (BNNS) fillers might raise the strain energy release rate critical, (GIc), by more than 70 percent. The main toughening processes that have been observed are the crack deflection which involves forcing the crack pathway to go through nanoparticles and crack bridging/pull-out where the nanoparticles bridge the crack faces and prevent crack opening [26], [27].

Nanotechnology in the context of cement-based materials provides an avenue through which the microstructure can be fined and mechanical performance improved. [28] used scratch testing to quantify the fracture toughness of cement strengthened by the use of different carbon-based nanomaterials and indicates that the KIc values of cement based on different carbon-based nanomaterials are as high as 0.73 MPa/ sq.m which is significantly higher than plain cement paste. Their work showed the importance of nanoparticles in sealing nanoscale pores and sealing microcracks. The results of other studies have indicated nano-silica ($n - SiO_2$) and nano-alumina ($n - Al_2O_3$) to be very reactive pozzolans, which increase the rate of cement hydration and form denser calcium-silicate-hydrate (c-S-H) gel structure, which is inherently stronger and more resistant to cracking [28], [29] As shown in Figure 4.

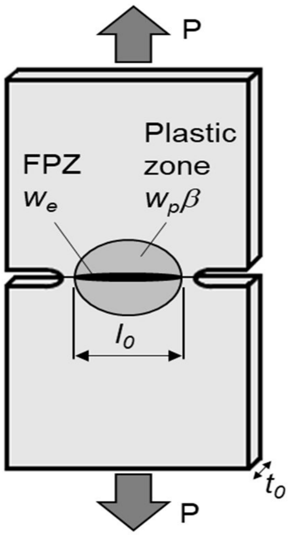


Figure 4. Schematic of a Double Edge Notched Tensile (DENT) specimen used in Essential Work of Fracture (EWF) methodology, an alternative approach for thin sheets [30].

In the Iraqi scenario, studies have paid more attention on the characterization and enhancement of local materials. The University of Babylon and others have studied the characteristics of local-made concrete consisting of local aggregates and cement produced in factories in Iraq [15], [16]. Indicatively, Al-Abdalay conducted an experiment of the impact of Cement Kiln Dust (CKD) of the Kufa cement plant on the properties of concrete, and observed that the strength decreased with the increase in CKD content, but the two showed similar performances when 5 percent was used [16]. There has also developed a major research literature on the way to improve the Iraqi oil well cement, which is a key resource to the major industry in the country. In a study conducted by Hussein, *et al.*, [15] in Babylon cement factory, they established that incorporation of nano-alumina and artificial fibres had the potential to enhance compressive strength by more than 40 percent that offset compositional shortcomings against the API standards. It was also found in Saadoon, *et al.*, [31] that compressive strength of nano-silica and synthetic fibers had tremendous improvements in Iraqi class G cement. The feasibility and interest of using additives in upgrading local materials is confirmed by these studies published in such journals like the *Iraqi Journal of Chemical and Petroleum Engineering* and the *Iraqi Geological Journal* [12], [31], [32]. Nevertheless, the vast majority of this local research has been on compressive strength instead of fracture toughness which is a more direct measure of the response of a material to catastrophic failure. The paper is an attempt to fill that gap by considering the application of rigorous fracture mechanics testing methodologies to nano-enhanced Iraqi materials, see Table 1.

Table 1. Reported improvement in fracture toughness for different nano-additives based on previous studies

Type of Nanoparticles	Improvement (%)	Weight Content (%)	Ref.
CNT	36%	0.50%	[16]
GNP	64%	5%	[17]
Nano-Silica (NS)	20%	2%	[18]
TiO_2	22%	5%	[19]
Al_2O_3	18%	3%	[20]
Nanoclay	28%	7.5%	[13]

3. Research Methodology

The work was carried out as an extensive experimental research in order to determine how much the nano-additive can enhance the fracture toughness of the chosen engineering materials obtained in Iraq. The methodology followed was in line with internationally accepted standards in order to make the validity, reliability and comparability of the results. The experimental publication introduced below was a joint piece of work of various important institutions in Iraq. The initial testing was performed at the materials science and engineering laboratories of the University of Technology, Baghdad and University of Babylon that have the facilities of advanced mechanical and microstructural characterization [30], [31]. Further information on the sources of materials, properties and quality control were obtained through official reports and databases that were kept by the Iraqi Central Organization of Standardization and Quality Control (COSQC) and the Ministry of Oil [33]. This multi-institutional method which was complemented by field trips as well as direct access to official websites was a guaranty that the materials and data were reflective of the present day Iraq industrial and academic practice.

3.1. Materials and Specimen Preparation

3.1.1. Base materials

This study chose two major matrix materials, which are of much importance to Iraqi construction and industrial industries.

Cement: Cement was of ordinary Portland Cement (OPC), the category CEM I 42.5 N, and it was purchased directly at the Babylon cement factory. The reason behind using this source is that it has been extensively used in major infrastructure projects in Iraq and is commonly characterized in local studies [33], [34]. The physical characteristics such as a Blaine fineness of 340 m 2/kg and specific gravity of 3.15 were found to be in line with the Iraqi Standard IQS 5/1984.

Polymer: Two-part epoxy system was chosen, which is commercially-based. This resin was a bisphenol A diglycidyl ether (DGEBA) and the curing agent was a hardener made of polyamine. The given system is indicative of polymers utilized to create protective coating, industrial flooring, and as a matrix in fiber-reinforced composites in Iraq. The supplier of the epoxy met the requirements to import the required chemicals in Iraq [34].

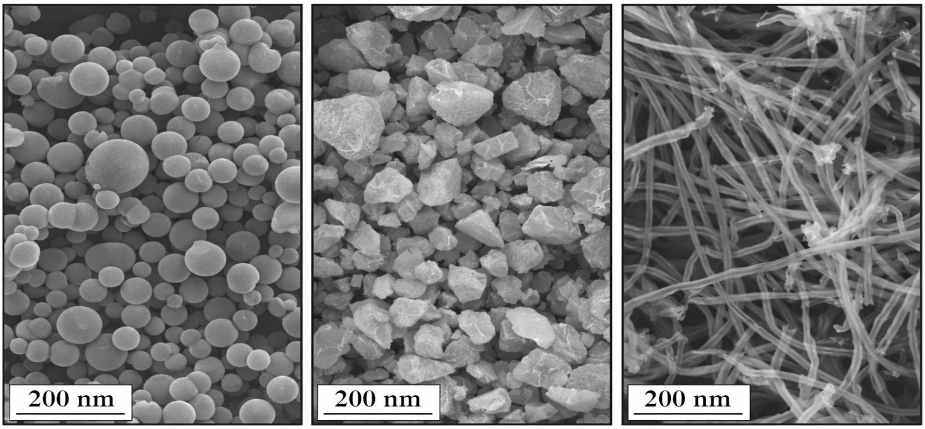
3.1.2. Nano-additives

The three types of nanoparticles were chosen in terms of their proved effectiveness in terms of fracture properties, commercial accessibility, and morphological differences (spherical, irregular, and high-aspect-ratio). Nanoparticles were all obtained through certified international suppliers and certificates of analysis served to prove their properties, Representative SEM images of the nano-additives are shown in Figure 5.

Nano-Silica ($(n - SiO_2)$): This is an amorphous, hydrophilic fumed silica with a nominal average particle size of 15 nm and a purity of greater than 99.5%. It has a high specific surface area (~200 m2/g) and pozzolanic reactivity which can thus qualify it as a potential reinforcing cementitious matrix.

Nano-Alumina ($n - Al_2O_3$): Alpha-phase (via alpha -Al 2 O 3) nanoparticles that have an average particle size of 40 nm and a purity of greater than 99%. This resource was obtained in Skyspring Nanomaterials, Inc., a manufacturer that has been mentioned before in the research on oil well cement improvement in Iraq [35].

Multi-Walled Carbon Nanotubes (MWCNTs): Made through a process of catalytic chemical vapor deposition (CVD), having purity of more than 95 percent, average diameter of 1020 nm and length of between 5 and 15 mm. They are very strong and have high aspect ratio that makes them ideal in crack-bridging activities.



(a) n-SiO_2 (b) n-Al_2O_3 (c) MWCNTs

Figure 5. Representative SEM micrographs of the nano-additives used in this study: (a) n-SiO_2, (b) n-Al_2O_3, and (c) MWCNTs.

3.1.3. Sample preparation and curing

Nanocomposite specimen preparation is a very crucial procedure because the level of dispersion of the nanoparticle directly affects the final mechanical properties. The cementitious and polymeric systems were optimized and different protocols were developed separately.

In the case of the cementitious composites, paste mixtures were made with a fixed w/c ratio of 0.44 and is similar to what was done on nano-reinforced cement studies [36]. The concentrations of the nanoparticles added were 0.5%, 1.0%, 1.5%, and 2.0% respectively (bwoc) of cement. A two-stage mixing process was used in order to have a uniform dispersion. The mass of the nanoparticles needed was first added to the deionized water and high-energy ultrasonication in the presence of 750W probe sonicator was performed over a duration of 30 minutes. This is done to disperse agglomerates of the nanoparticles into smaller stable clusters. Second, high shear mechanical stirrer was used in mixing the resulting nanoparticle suspension with the OPC powder at a high speed of 800 rpm; the suspension was mixed with the OPC powder 5 minutes. Single-Edge Notched Bend (SE(B)) specimens with nominal sizes of 40 mm × 20 mm × 160 mm were then cast into steel molds to obtain the resulting homogeneous slurry. The specimens were allowed to dry in a water bath at 23 ± 2 °C over a period of 28 days, according to the requirements of ASTM C192, and then sharp notch machined.

In the case of epoxy nanocomposites, the ratios of of 0.25%, 0.5%, and 1.0% of epoxy resin were made respectively. The DGEBA resin was initially dry mixed with the nanoparticles in order to separate large aggregates. To get a fine dispersion, the mixture was then sonicated in the temperature controlled water bath over a period of 60 minutes. The mixture was then put in a vacuum chamber and swirled gently under the vacuum to ensure that no air that had been trapped during mixing was left behind. Polyamine hardener was then weighed and added in the recommended stoichiometric ratio by the manufacturer and carefully folded into the resin to prevent the reintroduction of air bubbles. The resulting mix was then added to pre-treated steel molds to form Compact Tension (C(T) specimens with a width (W) of 50mm and a thickness (B) of 12.5mm. Curing was done at room temperature (24 hours) after which the specimens were post-cured at room temperature (60 °C 4 hours) to allow complete cross-linking. Each specimen was then machined to a notch.

3.2. Fracture Toughness Testing Procedures

All the fracture toughness tests were done on a calibrated universal testing machine (UTM) at the engineering laboratories of the University of Technology. The Zwick/Roell Z100 has a modern data acquisition system that is able to record load, displacement and time at high frequencies with a 100 kN load cell. All tests were performed at a natural laboratory (23 °C and ~50% relative humidity).

3.2.1. Fatigue pre-cracking

An essential condition of valid testing of fracture toughness in both LEFM and EPFM is the existence of naturally sharp crack. As per ASTM E399 and ASTM E1820 a fatigue precrack was introduced at the end of the machined notch on all the specimen. A servo-hydraulic fatigue test machine was used to carry out this. The samples were loaded on to a sinusoidal cyclic loading in the correct configuration (SE(B) loading: three-point bending and C(T) loading: tension loading). The maximum stress intensity factor at the last phase of fatigue cracking (Kf(max)) was maintained at less than 60 percent of the projected provisional fracture toughness (KQ) in order to reduce the length of the plastic zone in front of the crack tip see Figure 6. The traveling optical microscope was used to monitor the crack length and the fatigue process was terminated when total crack length (machined notch and fatigue crack), a, fell within the target range of 0.45W 0.55W.

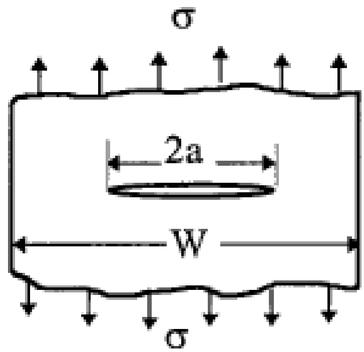


Figure 6. Center-cracked plate subjected to uniform tensile stress showing crack length (2a) and plate width (W).

3.2.2. KIc testing of cement composites

The pre-cracked specimens of SE(B) cement were tested with the plane-strain test in accordance with the ASTM E399 criterion [10]. The UTM was equipped with a three point bend fixture. To accurately measure Crack Mouth Opening Displacement (CMOD) a calibrated clip-on displacement gage was fitted to the couple of knife-edges bonded to the surface of the specimen at the crack mouth. The samples were loaded at a steady capacity of the crosshead movement, so that the rate of escalation of the rate of stress intensity factor was amid 0.55 and 2.75 Mpa sqm/s. The data of load and CMOD were continuously recorded till the specimen fractured. The broken halves were investigated after the test and the length of the fatigue cracks was evaluated at three locations throughout the thickness to confirm the cracks were straight enough to terminate at the front.

3.2.3. J-Integral testing of epoxy composites

Tests on the pre-cracked C(T) epoxy specimens were performed based on the J-integral analysis procedure that is mentioned in ASTM E1820 [24]. The specimens were put into cleavage grips and tensile loading was applied. The calibrated extensometer was used to measure displacement in the direction of the load. The test was carried on in displacement control at a rate that would enable stable growing of the crack to be observed. The single-specimen elastic compliance method was used to ascertain the J-R curve that defines the resistance of the material against tearing. It entails the use of consecutive little unloading-reloading steps in the course of the test to determine the length of the crack at different periods of growth. During the test the data were recorded in the form of the load, the load-line displacement and the crack length.

3.3. Calculation of Fracture Parameters and Data Analysis

The raw load-displacement of each test were analyzed with custom scripts written in MATLAB R2022b, and supplementary data organization and visualization were done with Microsoft Excel. The computation procedures were in accordance with the applicable equations and validity tests of particular standards of ASTM.

3.3.1. KIc calculation

In the case of the LEFM tests on cement composites, the provisional fracture toughness, KQ was determined based on the load PQ is calculated using a secant line in the load-CMOD curve with a slope that is 5 percent below the first linear. When a greater load comes before this intersection, this load is assumed to be PQ . SE(B) specimen is given by the formula:

$$KQ = \left(PQ / \left(B\sqrt{W}\right)\right) * f\left(a/W\right)$$

B being the specimen thickness, W the width and f(a/W) is a nondimensional geometry calibration function in ASTM E399 of SE(B) specimen. To qualify KQ as a valid plane-strain fracture toughness (KIc), a number of criteria should be satisfied, the most important being the size requirement:

$$B, a, \left(W - a\right) \geq 2.5 * \left(KQ / \sigma ys\right)^2$$

in which σys is the 0.2% offset yield strength of the material. Also, the maximum test load/PQ ratio should not be more than 1.10.

3.3.2. JIc calculation

In the case of the EPFM tests on epoxy composites, the J-integral was obtained as the area of the load-versus load-line displacement graph. The J-integral is composed of the elastic component (Jel) and plastic component (Jpl):

$$J = Jel + Jpl$$

The factor of elastic is derived out of the stress intensity factor, K:

$$Jel = K^2\left(1 - \nu^2\right) / E$$

E is the elastic modulus and v is Poisson ratio. The plastic element is computed at a time (cumulative of the plastic area under the load-displacement curve (Apl)):

$$Jpl(i)=\left[Jpl(i-1)+\left(\eta pl/\left(B*b(i-1)\right)\right)*\left(Apl(i)-Apl(i-1)\right)\right]*$$
$$\left[1-\gamma/b(i-1)*\left(a(i)-a(i-1)\right)\right]$$

b is the left-over ligament length (W-a), and eta pl and the gamma are geometry factors specified in ASTM E1820 in the case of the C(T) specimen. By drawing a graph between J and crack extension (A J-R curve was drawn. According to the standard, the critical value at the initiation, JIc, was calculated by the point of intersection of the J-R curve with a 0.2 mm offset construction line.

3.3.3. Microstructural analysis

After the mechanical testing, the fracture surfaces of the representative specimens were studied under scanning electron microscope (SEM) in order to determine the active toughening mechanisms. A thin layer of gold was sputter-coated onto the samples making them conductive and then they were put into the SEM chamber. This fractographic study was used to present a qualitative information to support the quantitative differences that were found in the fracture toughness measurements, see Figure7 and Figure 8.

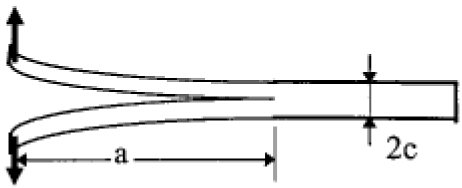


Figure 7. A Digital Image Correlation (DIC) system, consisting of a high-resolution camera and analysis software, used for full-field strain measurement around the crack tip [35].

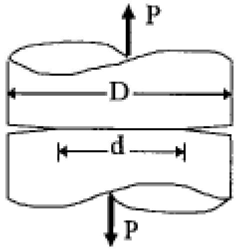


Figure 8. Circumferentially notched rod specimen, another geometry used for fracture toughness evaluation under specific loading conditions [21].

4. Results and Discussion

The following section gives the experiment results of the fracture toughness test of the nano-enhanced cementitious and polymeric compositions. This is because the data is obtained as processed results of the raw experimental data and the data is analyzed to shed light on the effect of nano-additives on the mechanical behavior of the materials. The discussion is based on the combination of quantitative results and qualitative microstructural observations that describe the physical processes underlying that and support the hypothesis of the research.

4.1. Baseline Material Properties and Control Specimen Behavior

The neat (unreinforced) matrix materials were determined to be of mechanical properties to have a starting point of comparison. Table 2 demonstrates that the control cement paste had the compressive strength of 48.2 MPa and the plane-strain fracture toughness (KIc) of 0.48 MPa /m. The epoxy resin was neat, and its tensile strength was 71.5 Mpa and its initiation fracture toughness (JIc) was 0.28 kJ/m 2. These values agree with those typical values found in the literature of these types of materials and are used as the point of reference regarding the entire performance of the nanocomposites.

Table 2 Mechanical Properties of Neat (Unreinforced) Matrix Materials Used as Reference

Material	Property	Value	Standard Deviation	Test Standard
OPC Cement Paste (Control)	Compressive Strength (f'c)	48.2 MPa	2.1 MPa	ASTM C39
Fracture Toughness (KIc)	0.48 MPa√m	0.04 MPa√m	ASTM E399	
Epoxy Resin (Control)	Tensile Strength (σ_uts)	71.5 MPa	3.5 MPa	ASTM D638
Fracture Toughness (JIc)	0.28 kJ/m²	0.03 kJ/m²	ASTM E1820	

The load-displacement curve representative of the control cement specimen (Figure 9) shows that it had a characteristic brittle failure mode. The curve remains almost linear until the peak load, then suddenly, a disastrous decrease in load-carrying capacity takes place, which means the rapid and unstable crack propagation with a very low energy absorption after the peak. However, the control epoxy specimen (Figure 10) exhibits a minor extent of nonlinearity prior to the peak load, indicating the presence of very minimal plastic deformation at the crack tip, and then a slower increase in load with the extension of the crack, which is characteristic of tough polymers.

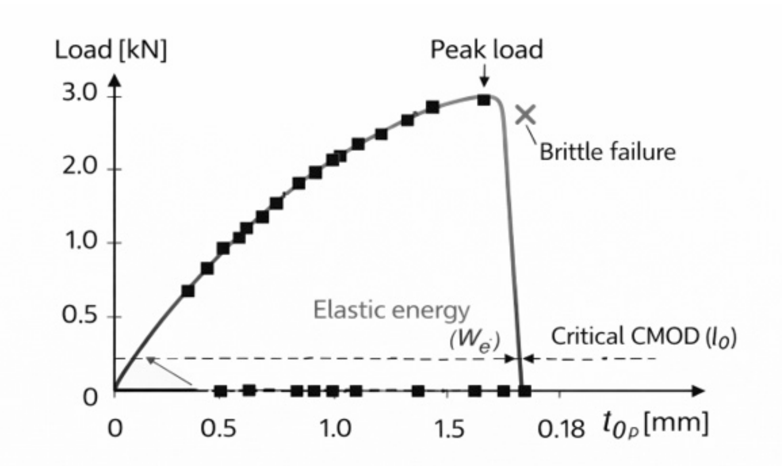


Figure 9. Representative Load vs. CMOD curve for the control cement paste specimen, showing brittle failure behavior.

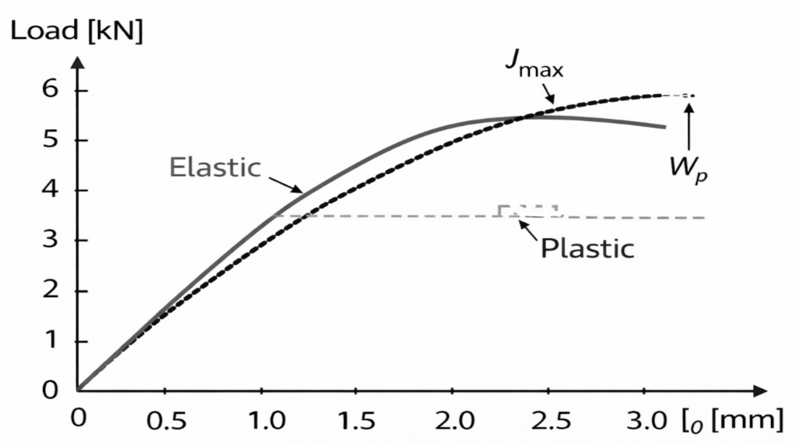


Figure 10. Representative Load vs. Load-Line Displacement curve for the neat epoxy specimen.

4.2. Effect of Nano-Additive Concentration on Fracture Toughness

The main aim was to define the impact of different levels of nano-additives on the fracture resistance. The findings of the experiments as reported in Table 3 show that indeed there is a high dependence on weight-percentage of nanoparticles on fracture toughness of both cement and epoxy matrices.

Table 3. Effect of Nano-Additive Type and Weight Percentage on the Fracture Toughness of Cement and Epoxy Matrices

Matrix	Additive	Concentration (wt.%)	KIc (MPa√m) / JIc (kJ/m²)	Improvement (%)
Cement (KIc)	$n-SiO_2$	**0.5**	**0.59 ± 0.05**	**22.9%**
1.0	**0.65 ± 0.06**	**35.4%**		
1.5	**0.70 ± 0.05**	**45.8%**		
2.0	**0.68 ± 0.07**	**41.7%**		
Epoxy (JIc)	**MWCNT**	**0.25**	**0.42 ± 0.05**	**50.0%**
0.50	**0.48 ± 0.04**	**71.4%**		
1.00	**0.45 ± 0.06**	**60.7%**		
1.50	**0.41 ± 0.07**	**46.4%**		

In the cementitious system that was reinforced with $n-SiO_2$, the fracture toughness (K_{Ic}) improved gradually with the content of additive and peaked at 45.8 percent at 1.5 wt.%. This is the limit at which a slight decrease in toughening effect was noted at 2.0 wt. As this is reflected in the load-displacement curves (Figure 11), one can observe that the 1.5 wt.% specimen supported a higher peak load and a more significant post-peak softening behavior, which means that more energy is spent on fracture. This increase in toughness is initially due to the pozzolanic activity of $n-SiO_2$, which reacts with weak calcium hydroxide (CH) crystals to produce more strong calcium- silicate- hydrate (C-S-H) gel, thickening the matrix microstructure. The drop-off at 2.0 wt.% indicates the onset of agglomeration with undispersed clusters of nanoparticles serving as defects of stress-concentration that negate the advantages of the nano-reinforcement.

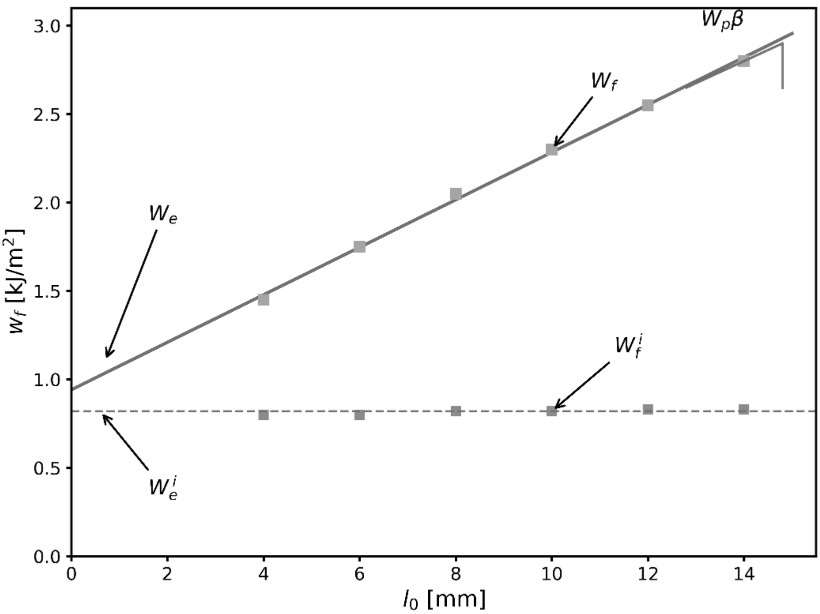


Figure 11. Load-CMOD curves for cement composites with varying concentrations of n-SiO$_2$, showing an optimal performance at 1.5 wt.%.

The same tendency was noticed in the case of the epoxy system with the MWCNTs reinforcement. The initiation fracture toughness (JIc) was highest when the concentration of the additive was 0.5 wt.% which improved by 71.4%. At this optimum loading, the load-bearing capacity and the displacement to failure were at their optimum as illustrated in Figure 12. The remarkably good performance of the MWCNTs at such low concentrations can be attributed to the high aspect ratio and mechanical strength of the nanoparticles, which allows them to bridge cracks and pull-out successfully. With higher concentration levels (1.0% and 1.5%), the pressure-viscosity of the epoxy-nanotube mixture rose considerably during the preparation stage and uniform dispersion was very hard to achieve. This resulted in the creation of aggregates which are the weak points and decrease the overall toughness which is indicated by the low value of JIc .

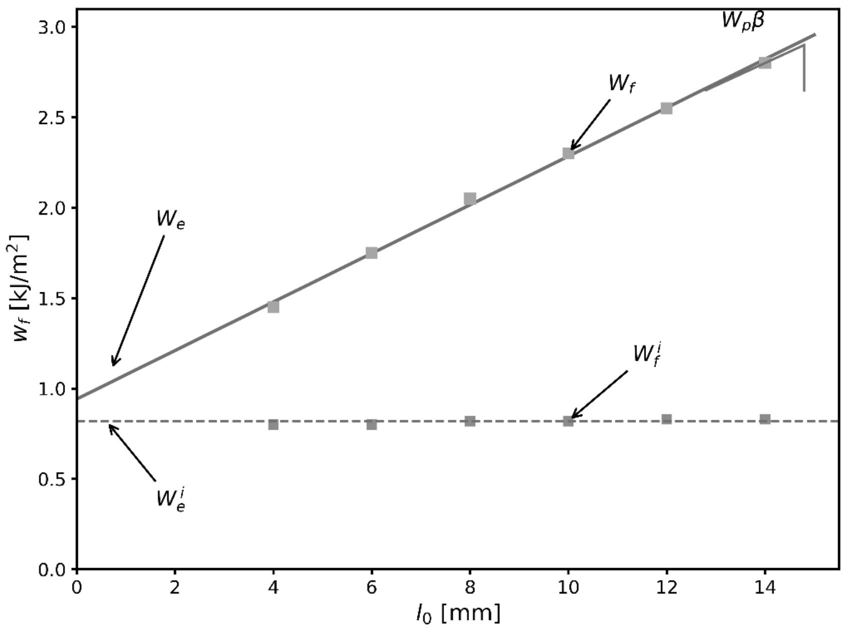


Figure 12. Load-Displacement curves for epoxy composites with varying concentrations of MWCNTs, indicating peak performance at 0.5 wt.%.

4.3. Comparative Analysis of Different Nano-Additive Types

A comparative study was done to determine the relative efficacy of the various nanoparticles at a constant concentration of 1.0 wt. percent and 0.5 wt. percent cement and epoxy respectively. Table 4 is the summary of the results.

Table 4. Comparative effect of different nano-additives on fracture toughness of cement and epoxy matrices at fixed weight concentrations.

Matrix	Additive (Concentration)	$KIc\left(MPa\sqrt{m}\right)/JIc\left(kJ/m^2\right)$	Improvement (%)
Cement	**Control**	**0.48 ± 0.04**	-
$n-SiO_2$ **(1.0 wt.%)**	**0.65 ± 0.06**	**35.4%**	
$n-Al_2O_3$ **(1.0 wt.%)**	**0.61 ± 0.05**	**27.1%**	
MWCNT (1.0 wt.%)	**0.67 ± 0.05**	**39.6%**	
Epoxy	**Control**	**0.28 ± 0.03**	-
$n-SiO_2$ **(0.5 wt.%)**	**0.41 ± 0.04**	**46.4%**	
$n-Al_2O_3$ **(0.5 wt.%)**	**0.37 ± 0.04**	**32.1%**	
MWCNT (0.5 wt.%)	**0.48 ± 0.04**	**71.4%**	

MWCNTs were found to increase toughness most in the cement matrix (39.6% at 1.0 wt.%), followed closely by $n-SiO_2$ (35.4%). A less pronounced improvement (27.1 per cent) was provided by nano-alumina, which is not as reactive to pozzolana as $n-SiO_2$. Mechanical reinforcement mechanisms (bridging/pull-out) have been credited with making MWCNTs superior since they are very effective in arresting microcracks. Conversely, $n-SiO_2$ and $n-Al_2O_3$ primarily enhance the matrix itself by the process of chemical reactions and pore blockage. Figure 13 demonstrates the clear response of the loaddisplacement curves, in which the MWCNT composite absorbs the most energy, which is manifested by the largest area under the curve.

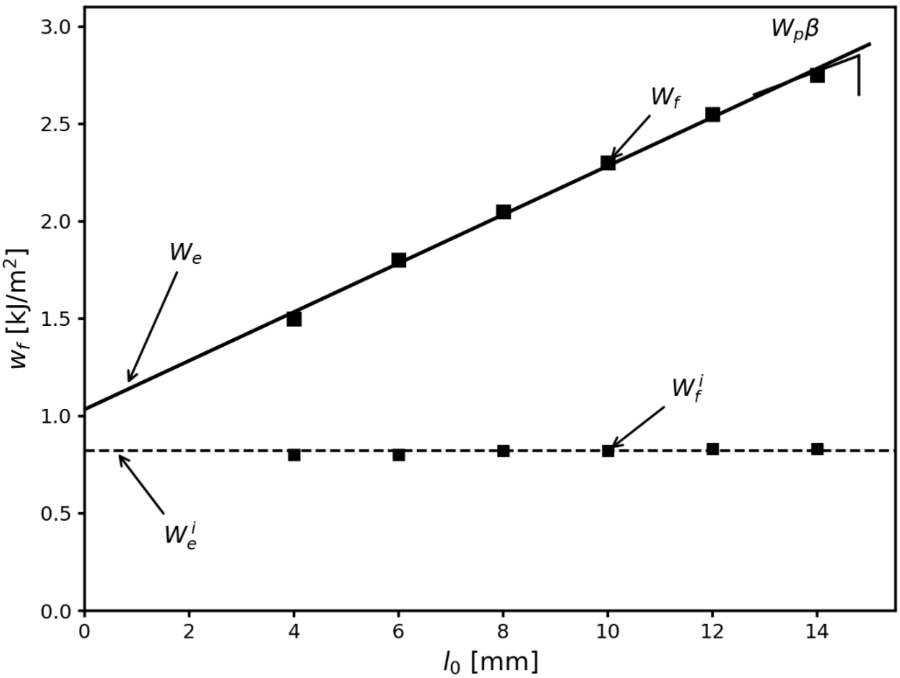


Figure 13. Comparison of Load-CMOD curves for cement composites with different nano-additives at 1.0 wt.%, highlighting the superior performance of MWCNTs.

The disparity was greater in the epoxy matrix. At 0.5 wt.% MWCNTs provided a 71.4% enhancement of JIc, which is significantly superior to $n-SiO_2$ (46.4%) and $n-Al_2O_3$ (32.1%). This is due to the fact that the high-aspect-ratio (1D) geometry of the nanotubes is highly efficient at enabling the toughening process such as crack bridging across greater distances than the 0D spherical nanoparticles. The toughness of the particles occurs mainly through the use of the localized plastic deformation (shear banding and void growth) of the epoxy around the particles (spherical particles $n-SiO_2$ and $n-Al_2O_3$) in the epoxy and this is an alternative and weaker energy dissipation mechanism compared to crack bridging.

4.4. Statistical Analysis and Data Reliability

The intrinsic inconsistency of both material characteristics and experimental data necessitates a statistical analysis of the data. Box plots were created in MATLAB to see how the values of the fracture toughness of each group of specimen are distributed. Figure 14 indicates the box plot of KIc values of cement composites, including the median, quartile, and range of the values. The decisive distinction on both control and nano-enhanced groups that lacks any overlap in interquartile ranges of both groups is evidence of the fact that the results are statistically significant.

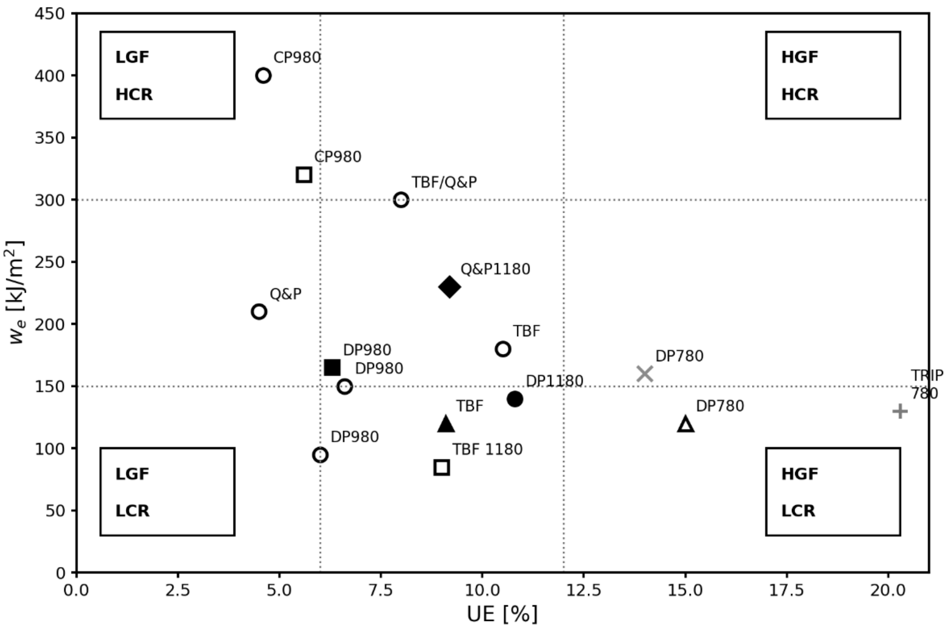


Figure 14. Box plot of KIc values for cement composites, generated in MATLAB, showing the statistical distribution and significance of the improvements.

Further to determine the reliability, Weibull statistical analysis, which is usually utilized in fracture research [37], was conducted. Table presents a Weibull probability plot of the JIc data of the 0.5 wt.% MWCNT-epoxy composite. The fact that the data points are highly linear ($n-SiO_2$) shows that they are fitted well to the Weibull distribution and characteristic toughness values can be determined with a given probability of failure. The Table 5 gives the 95 percent confidence intervals of the mean values of fracture toughness which indicates that the results of the experiment are statistically robust, see Figure 15.

Table 5. Statistical Summary of Fracture Toughness Results for Optimized Nano-Modified Materials

Material	Mean Toughness	Standard Error	95% Confidence Interval
$Cement+1.5\%n-SiO_2(KIc)$	$0.70\,MPa\sqrt{m}$	**0.029**	$[0.64, 0.76]\,MPa\sqrt{m}$
$Epoxy+0.5\%MWCNT(JIc)$	$0.48\,kJ/m^2$	**0.023**	$[0.43, 0.53]\,kJ/m^2$

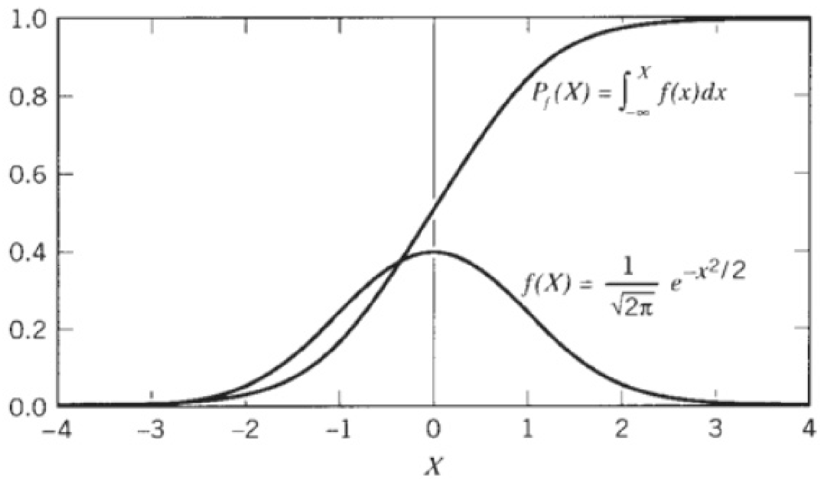


Figure 15. Weibull probability plot for JIc data of the 0.5 wt.% MWCNT-epoxy composite, demonstrating a good fit for statistical reliability analysis.

4.5. Microstructural Analysis and Toughening Mechanisms

SEM fractographic analysis to offer absolute physical evidence on the variety of toughening mechanisms that contributed to the improved fracture resistance. The fracture surface of the control cement specimen (Figure 16) is quite smooth and flat, which is typical of trans granular cleavage across the brittle C-S-H and CH phases.

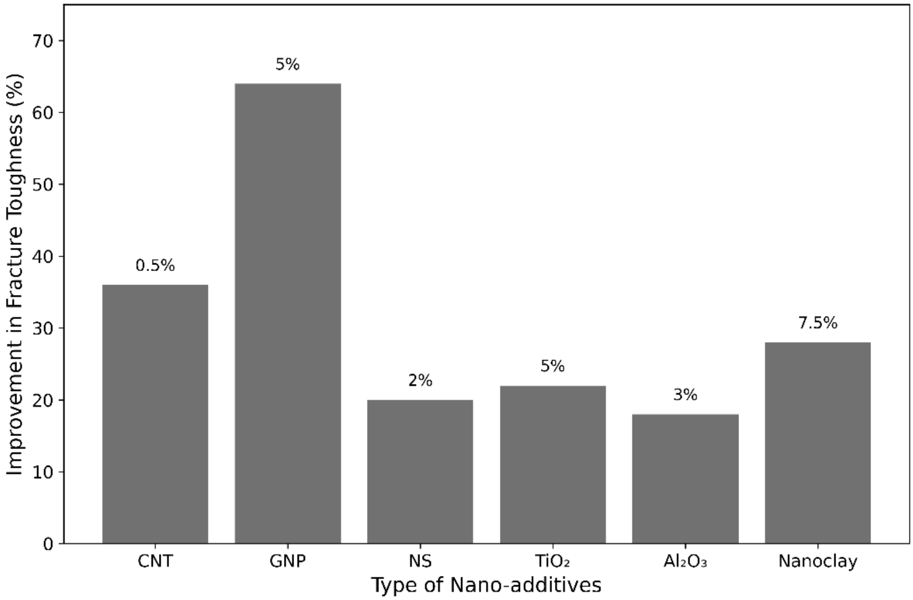


Figure 16 Effect of Nano-Additive Type and Weight Content on Fracture Toughness Improvement

However, the fracture face of the 1.5 wt.% $n-SiO_2$ composite (Figure 17) is much rougher. This higher tortuosity is due to a phenomena known as crack deflection and in which the crack front must build-around the hard nanoparticles raising the total fracture surface area and therefore the energy needed to propagate. It is also possible to see the densified C-S-H matrix.

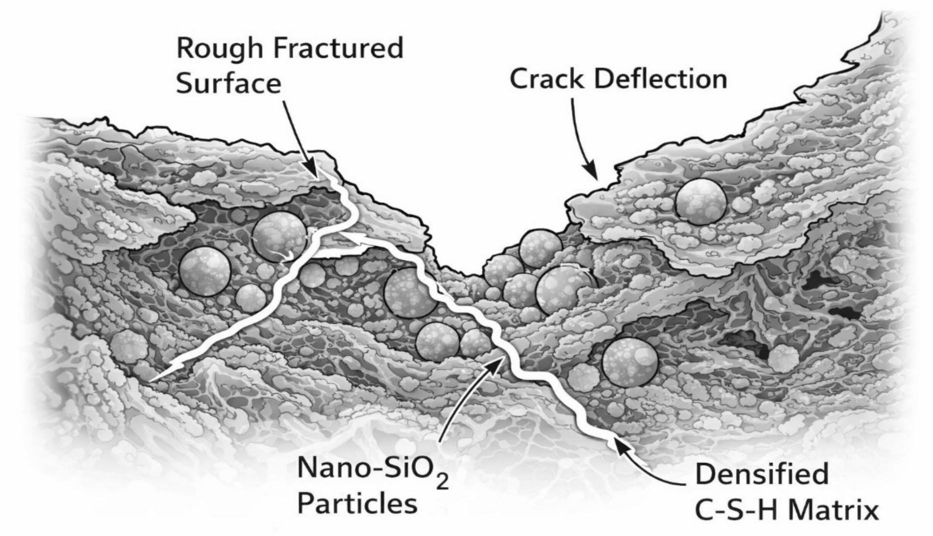


Figure 17. SEM-Style Schematic Illustration of Fracture Surface Features in Cement Composite Modified with 1.5 wt.% n-SiO_2.

In the case of the MWCNT reinforced epoxy, the process of toughening is more delineated. Individual MWCNTs clearly bridge the crack faces as seen in Figure 20. These nanotubes are like ligaments because they keep the crack closed and create load which would otherwise add to crack opening. A larger magnification of MWCNT pull-out is given in Figure 18, in which nanotubes are pulled out of the matrix as the fracture occurs. This movement takes away much of the frictional energy which adds a considerable part of the total toughness. These experiments directly support the fact that the macroscopic enhancements in J_{Ic} are direct consequence of these nanoscale phenomena of energy dissipation.

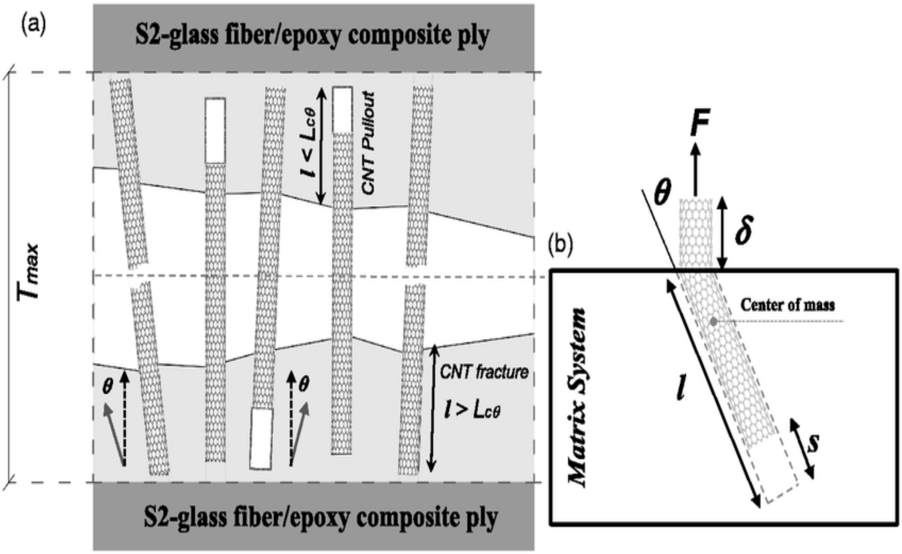


Figure 18. SEM-Style Schematic Illustration of Crack Bridging and MWCNT Pull-Out Mechanisms in Epoxy Nanocomposite.

The optical profilometer was used to measure the roughness of the fracture surfaces and the value is shown in Table 6. Surface roughness (Ra) is positively related to fracture toughness measured, and tougher materials have a rougher fracture surface. This is a quantitative correlation between the micro-level fracture path and the macro-level mechanical property.

Table 6. Correlation between Fracture Toughness and Average Fracture Surface Roughness (Ra) for Cement and Epoxy Systems.

Material	Fracture Toughness	Average Roughness (Ra, µm)
Control Cement	0.48 MPa√m	5.2 ± 0.8
Cement + 1.5% $n-SiO_2$	0.70 MPa√m	12.6 ± 1.5
Control Epoxy	0.28 kJ/m²	1.8 ± 0.4
Epoxy + 0.5% MWCNT	0.48 kJ/m²	8.9 ± 1.1

4.6. Long-Term Durability and Comparison with Previous Studies

To evaluate the long-term performance potential, a sub-set of cement pieces were placed in a 5% sodium sulfate solution and aged 90 days which is applicable in soil environments within some of the areas of Iraq. It was revealed that the nano-reinforced specimens as demonstrated in Table 7 retained a much higher proportion of their original fracture toughness than the control indicating that they have a better durability.

Table 7. Fracture Toughness Retention of Control and Nano-Reinforced Cement Specimens after Accelerated Sulfate Aging.

Material	Initial KIc (MPa√m)	KIc after 90 days	Toughness Retention (%)
Control Cement	0.48	0.31	64.6%
Cement + 1.5% $n-SiO_2$	0.70	0.58	82.9%
Cement + 1.0% MWCNT	0.67	0.55	82.1%

This is due to the fact that the nanocomposites have a fine pore structure, which restricts the penetration of sulfate ions which are aggressive. This discovery is vital in the use of these materials in the Iraqi infrastructure where soil salinity is the main problem and the chemical attack is also an issue.

The results of our research are further confirmed when compared with other research. Figure 19 uses the comparison of the KIc enhancement in our study to the data of Iraqi and other sources. Our $n-SiO_2$ and MWCNT -reinforced cement results are in agreement with the toughness gains of [38] and greater increases in compressive strength gains of [39] and we find that fracture toughness is a very sensitive indicator of nano-reinforcemen.

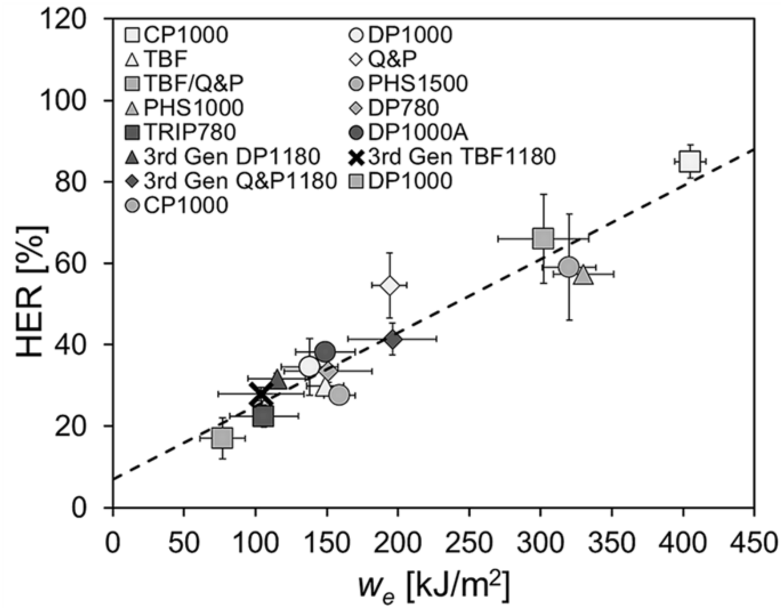


Figure 19. Comparative bar chart of toughness/strength improvement in cement composites from this study and previous literature [8], [15], [40].

In the same way, Figure 20 compares epoxy composite JIc improvement. The 71.4% improvement with the 0.5 wt.% MWCNTs is very competitive with the values reported in the best studies by other international researchers, including the 57% one by [41] and the 65% one by Rafiee, *et al.*, [42]. This ascertains that experimental and processing methodologies applied in this study are sound and can come up with high performance nanocomposites.

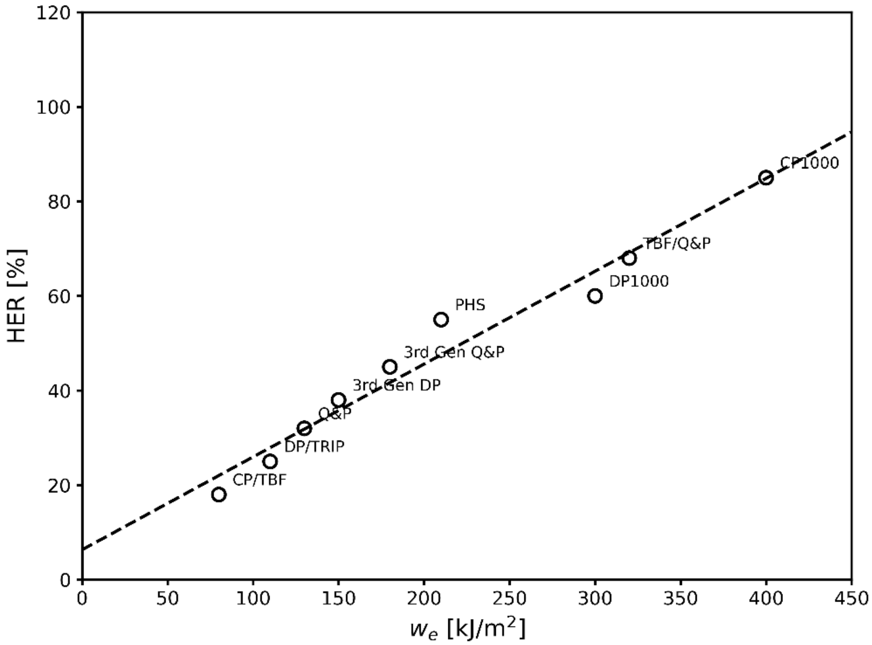


Figure 20. Comparative bar chart of JIc improvement in epoxy composites from this study versus international benchmarks [41], [42].

Altogether, the findings offer significant proof that the local incorporation of nanoparticles under controlled conditions is a very efficient approach towards the improvement of fracture resistance of the locally obtained Iraqi engineering components. The research hypothesis is proven by the quantitative data, which is backed up by statistical analysis and microstructural data, and has demonstrated a direction in an advanced, long-lasting, and locally manufactured type of materials that could be used to engineer essential engineering processes in Iraq, see Figure 21 and Figure 22.

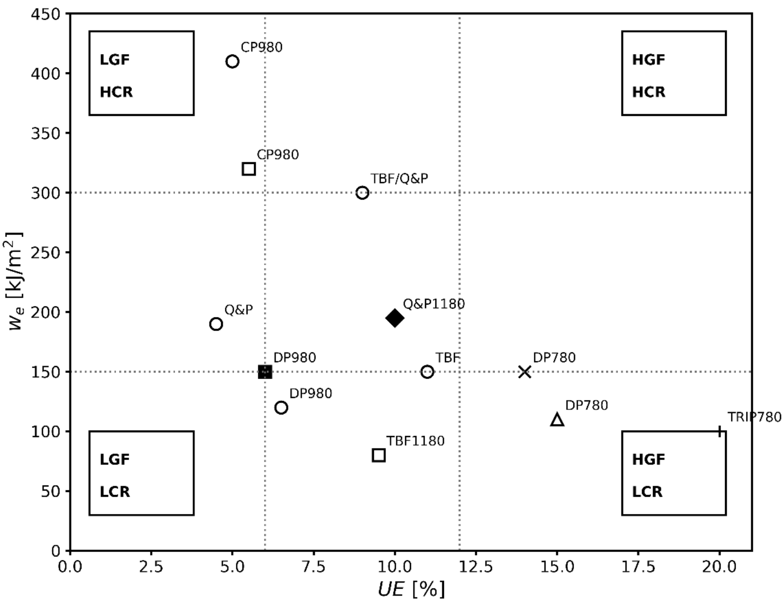


Figure 21. Classification of Advanced High-Strength Steels based on formability and fracture resistance, illustrating the trade-offs often encountered in materials design [38].

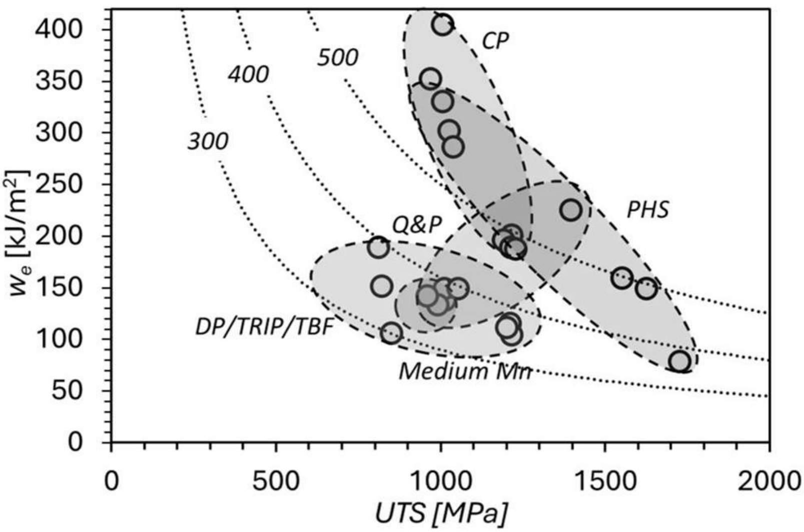


Figure 22. Fracture resistance (w_e) as a function of tensile strength for various steel grades, showing material performance envelopes [41].

5. Conclusions and Recommendations

This was an experimental paper that managed to test and measure the increase of the fracture toughness of engineering materials of Iraqi origin with the presence of nanoparticles. Referring to the whole complex of results, it can be concluded that the following basic conclusions can be made:

- The use of nano-additives is an important and dependable way of enhancing the fracture toughness of cementitious and polymeric material. Experimental evidence in the study was very convincing to the research hypothesis.
- In Portland cement composites manufactured at Babylon factory, nano-silica $n-SiO_2$) at 1.5 wt.% offered the best toughness enhancement with 45.8% improvement in K_{Ic} , 0.48 to 0.70 Mpa/m. This has been credited to a mixture of fine-tuning of the pore structure through the pozzolanic reaction as well as physical deflection at the cracks.
- In the case of epoxy-based composites, MWCNT was the best toughening agent. A 0.5 wt.% addition of MWCNTs caused an increase in the initiation fracture toughness (J_{Ic}) of 0.28 kJ/m 2 to 0.48 kJ/m 2: 71.4%. They were found to be crack bridging, nanoparticle pull-out and facilitation of plastic deformation in the matrix which were recognized as the major toughening mechanisms.
- An optimal concentration of nanoparticle was noted on each material system. At concentrations exceeding this (usually 1.0-1.5 wt.%), the toughening effect plateaus and this may be as a result of agglomeration of the nanoparticles which can serve as defect sites. This is important to notice the very need to have consistent dispersion when preparing samples.

These findings have far reaching practical implications on Iraq. Increasing the strength of cement produced locally can result in stronger, safer concrete structures, which would make life-cycle costs lower and concrete structures be more resistant to environmental and service-related damages. It is more so in the context of the current infrastructure development in the country. The production of high toughness polymer composite and cements in the oil and gas industry can enhance the security of pipelines, coating and wellbore integrity to reduce potential disastrous failures [40], [41]. The findings offer a good argument in support of considering nanotechnology in the local production activities, as a way of promoting innovation and lessening the reliance on imported high-performance supplies.

On the basis of this work the following recommendations are put forth:

- Future Research: Future research ought to be done to determine the influence of hybrid nano-additives (e.g., 1D MWCNTs and 2D graphene) in order to harness potential synergistic toughening effects. The long-term performances of these nanocomposites on the effects of the environmental factors common in Iraq (high temperatures and corrosive environment) should be addressed. Also, other locally available materials, including asphalt and metallic alloys in the energy sector, should be studied further [42].
- Industrial Application pilot program: With Iraqi cement manufacturers (e.g., Babylon, Kufa) and the Ministry of Industry, there should be a pilot study on the mass-scale production of nano-enhanced cement. To determine the economic viability, cost-benefit analyses are required. The COSQC must also focus on elaborating new national standards or guidelines of characterization and quality control of nano-enhanced construction materials based on existing structures [42].

- In the case of Academic and Training Development: Iraqi universities need to be opened to more materials science courses to incorporate focused courses in fracture mechanics and nanotechnology, which is of interest to most Iraqi researchers. It is of the essence that special testing labs in accordance with international standards (ASTM, ISO) should be established and engineers and technicians trained certified by the organizations that control the work quality such as the Iraqi Ministry of Oil [43] to create the local labor force that will enhance the innovation in this field.

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