

Probabilistic Model for Flexural Strength of Cementitious Materials Containing CNTs

M. Ramezani¹, Y.H. Kim², Z. Sun³

¹Department of Civil and Environmental Engineering, University of Louisville, Louisville, KY, USA, mahyar.ramezani@louisville.edu

²Department of Civil and Environmental Engineering, University of Louisville, Louisville, KY, USA, young.kim@louisville.edu

³Department of Civil and Environmental Engineering, University of Louisville, Louisville, KY, USA, z.sun@louisville.edu

Abstract

The bond between carbon nanotubes (CNTs) and cementitious materials is the key characteristic for predicting the flexural strength. However, the dispersion quality dominantly changes bond mechanisms. A probabilistic approach can benefit a robust model development to capture various relations. This study proposes a probabilistic model based on the deterministic Kelly-Tyson theory to predict the flexural strength of CNT reinforced cementitious materials. The proposed model considers the influences of multiple experimental variables and their interactions on CNT dispersion quality and the flexural strength. A Bayesian methodology is adopted to calibrate the unknown model parameters and their statistical uncertainty using extensive experimental test results from the literature including the authors' work. The proposed model can be reliably used to predict the flexural strength with reasonable accuracy.

Keywords: Carbon nanotubes; Cementitious composites; Flexural strength; Probabilistic model; Kelly-Tyson theory

1 Introduction

Carbon nanotubes (CNTs) may significantly improve the flexural strength of cementitious materials because of their unique physical and mechanical properties (Brown *et al.*, 2019; Hajilounezhad and Maschmann, 2018; Hajilounezhad *et al.*, 2019). For example, CNTs possess outstanding elastic modulus and tensile strength of around 1 TPa (Demczyk *et al.*, 2002) and 30 GPa (Thostenson and Chou, 2003), respectively. To yield the highest flexural strength, the key factor is the bond strength between CNTs and the cementitious matrix which is dominantly governed by CNT dispersion quality. The dispersion quality, itself, depends on multiple factors such as physical characteristics of CNTs, dispersion procedure and mix proportions. Because of these complexities, there is a concern that CNTs might not disperse well within the cement matrix. As a result, inconsistent improvement or even degradation in the flexural strength of the composite has been reported in literature (Ramezani *et al.*, 2019a). Therefore, it is vital to understand the relationship between CNT dispersion quality and the mechanical properties of the composite in order to facilitate their application in the construction industry.

The bond mechanism between CNTs and the hosting matrix starts to engage when the stress is exerted. Therefore, the physical characteristics of CNTs are highly important for both the dispersion quality and the bond mechanism. If CNTs are well dispersed within the matrix, their high aspect ratio (length-to-diameter ratio) along with their nanoscale sizes reduces the distance between adjacent nanotubes, delaying the crack propagation. On the other hand, CNT high aspect ratio also makes them prone to agglomerate, reducing their reinforcement potential (Kafi *et al.*, 2016). If CNTs lack proper dispersion, the frictional forces become minimal within the agglomerated CNTs and they easily debond from the matrix (Bakshi and Agarwal, 2011), degrading the flexural strength. To overcome this, various dispersion techniques have been employed to uniformly disperse nanomaterials including CNTs within the cement matrix (Ramezani *et al.*, 2019a); amongst which an ultrasonication procedure is the most common practice (Ramezani *et al.*, 2016; Sadeghi-Nik *et al.*, 2017). Despite extensive research efforts, the contribution of CNTs to the flexural strength of cementitious materials is not yet fully understood. Therefore, in

the authors' previous work, extensive literature data was analyzed to identify the interactions between multiple experimental variables affecting the mechanical properties including the flexural strength (Ramezani *et al.*, 2019a). Then, three empirical expressions were introduced to include the identified interactions which enabled the prediction of the mechanical properties of CNT-cement nanocomposites within 20% error of the experimental test data using a simple regression model (Ramezani *et al.*, 2019b). However, the proposed regression model has a limitation of the application: it does not reflect the bond mechanisms of CNTs affecting the flexural strength.

This study aims to develop a reliable prediction model for the flexural strength of cementitious materials (pastes and mortars) reinforced with CNTs. To this end, the Kelly-Tyson model (Kelly and Tyson, 1965) which is extensively used to predict the tensile strength of brittle and ductile materials containing CNTs is selected as the base model. The Kelly-Tyson model is then modified by adding CNT orientation, length and volume fraction factors. Finally, additional critical relations are added to include different mechanisms of CNTs within cementitious materials. Besides, because incorporating CNTs is correlated with high degree of uncertainty, a probabilistic approach is adopted to consider various sources of uncertainty to affect the flexural strength. The proposed model provides valuable information for engineers and designers to determine and optimize the most important variables and the interactions between them to achieve the desired flexural strength.

2 Modified Kelly-Tyson Model

Different theoretical models are used to predict the tensile/flexural strength of CNT reinforced composites (e.g., polymer, metal and ceramic) (Chen *et al.*, 2017; Lachman *et al.*, 2012; Zare, 2016). Most of these models, however, assume a homogenous dispersion and perfect adhesion between CNTs and the matrix, resulting in inaccurate predictions. Despite the very high importance of these factors in determining the flexural strength, very few researchers tried to include them in prediction models. Besides, concerning cementitious composites, CNTs might also influence the hydration process of cement which needs to be considered (Ramezani *et al.*, 2019b). Therefore, there are very few models available to predict the flexural strength of CNT-cement nanocomposites. To address this, the current study adopts the Kelly-Tyson model as the base model to consider the bond mechanisms of CNTs in cementitious materials. The Kelly-Tyson model is then modified to include the mechanisms of CNTs within cementitious materials.

For condition of equal elastic strain in fiber and matrix, the rule of mixtures is considered as the simplest way to model the tensile strength of composites reinforced with aligned continuous fibers (Agarwal *et al.*, 2017):

$$\sigma_c = \sigma_f v_f + \sigma_m (1 - v_f) \quad (1)$$

where σ_c is the ultimate tensile strength of the composite (MPa), σ_f is the tensile strength of the fiber (MPa), σ_m is the tensile strength of the matrix at the ultimate strain of the composite (MPa) and v_f is the volume fraction of the fiber (unitless). To consider the contribution of the semi-aligned short discrete fibers with lengths (L) smaller than the critical length, Eq. (1) can be modified to include the fiber length and orientation factors as follows (Kelly and Tyson, 1965):

$$\sigma_c = \frac{1}{6} \tau_i \left(\frac{L}{d} \right) v_f + \sigma_m (1 - v_f) \quad (2)$$

where τ_i is the interfacial shear stress (MPa) and d is the fiber diameter (mm). However, due to the very low volume fraction of CNTs used in cementitious materials ($v_f = 0.00004-0.00128$; based on the literature), the predicted flexural strength of CNT-cement nanocomposites using Eq. (2) is almost the same as the flexural strength of the matrix. The authors' preliminary study has shown that $v_f^{2/3}$ is appropriate to fit the experimental data. Therefore, the modified form of the Kelly-Tyson model to predict the flexural strength of CNT-reinforced cementitious materials can be expressed as follows:

$$\sigma_c = \frac{1}{6} \tau_i \left(\frac{L}{d} \right) v_f^{2/3} + \sigma_m (1 - v_f^{2/3}) \quad (3)$$

Note that the modified Kelly-Tyson model (Eq. (3)) assumes uniform CNT dispersion and perfect bonding between CNTs and cement matrix. Therefore, the flexural strength increases as the CNT aspect ratio ($AR = L/d$) and/or v_f increases. Nevertheless, experimental studies exhibit nonlinear behavior: the flexural strength increases as AR

and/or v_f increases up to certain limits, beyond which the flexural strength degrades. The interfacial shear stress can be estimated by the link between the Pukanszky and the Kelly-Tyson models (Lazzeri and Phuong, 2014).

3 Proposed Critical Relations

To understand the mechanisms of CNTs within cementitious materials, three critical relations were proposed in the authors' previous work (Ramezani *et al.*, 2019b) to capture the interactions between the important experimental variables to predict the mechanical properties including the flexural strength.

- 1) dispersion relation (η_D) to correlate CNT dispersion as a function of AR , CNT volume fraction in aqueous solution (v_{f-w}), superplasticizer-to-CNTs ($SP/CNTs$) ratio, and Ultrasonication Energy Indicator (UEI)
- 2) matrix relation (η_M) to include the interactions between water-to-cement (w/c) and sand-to-cement (s/c) ratios
- 3) hydration age relation (η_H) to consider the interactions between CNT concentration (κ) and hydration age of cement (t)

A brief description of these critical relations is provided in Table 1. More details are provided in the authors' previous study (Ramezani *et al.*, 2019b). The applicability of the proposed critical relations to predict the flexural strength is validated in Section 5.

Table 1. Critical relations affecting mechanical properties.

Critical Relation	Equation	Descriptions
η_D	$1 - \left[\frac{AR \times v_{f-w}}{UEI \times (SP/CNTs)} \right]$	If v_{f-w} or $AR = 0$ (no CNTs added), $\eta_D = 1$ (perfect dispersion). As v_{f-w} and/or AR increases, η_D gets smaller. As UEI and/or $SP/CNTs$ ratio increases, η_D gradually reaches 1. Beyond certain UEI and/or $SP/CNTs$ ratio, η_D gets constant.
η_M	$\frac{(1 + s/c)}{(w/c)}$	As w/c ratio decreases, the value of η_M increases. As s/c ratio increases, the value of η_M increases.
η_H	$\left(\frac{t}{4 + 0.85t} \right)^\kappa$	If CNT is not added ($\kappa = 0$ $c-wt\%$), $\eta_H = 1$. At low CNT concentrations, the influence of t on η_H is minimal. As κ increases, η_H reaches 1 at later ages (i.e., a higher value of t).

In this study, three critical relations (η_D , η_H , and η_M) are added to Eq. (3) to fit the model to the experimental test results as follows:

$$\sigma_c = \eta_D \eta_H \eta_M \left[\frac{1}{6} \tau_i(AR) v_f^{2/3} + \sigma_m (1 - v_f^{2/3}) \right] \quad (4)$$

In addition, a Bayesian methodology is adopted to formulate the proposed model in a probabilistic manner to consider different sources of uncertainty such as the statistical uncertainty in the estimation of the unknown model parameters, the model errors associated with the inaccurate form of the model and missing variables (Gardoni *et al.*, 2002) that might influence the flexural strength.

4 Formulation of the Probabilistic Model

The probabilistic approach is used based on the modified deterministic Kelly-Tyson model (Eq. (4)). Then, additional correction terms (herein, η_D , η_H , η_M) and model error are introduced to the deterministic expression to account for inherent bias and to capture the remaining variability in residuals, respectively (Gardoni *et al.*, 2002). The probabilistic model relates the quantity of interest (herein, the flexural strength; σ_c) to a set of measurable variables (defined as $\mathbf{x} = (x_1, x_2, x_3, \dots)$) such as AR , κ , UEI , $SP/CNTs$ ratio, w/c ratio, s/c ratio, t , etc. The probabilistic capacity model can be written as follows:

$$C(\mathbf{x}, \boldsymbol{\Theta}) = C(\mathbf{x}) + \gamma(\mathbf{x}, \boldsymbol{\Theta}) + s\varepsilon \quad (5)$$

where $C(\mathbf{x}, \boldsymbol{\Theta})$ is the capacity model of interest (herein, σ_c), $\mathbf{x}$ is a set of measurable variables, $\boldsymbol{\Theta} = (\theta, \sigma)$, $\theta = (\theta_1, \theta_2, \theta_3, \dots)$ denotes a set of unknown model parameters used to fit the data, $C(\mathbf{x})$ is the selected deterministic model, $\gamma(\mathbf{x}, \theta)$ is the correction term (herein, η_D , η_H , and η_M), s is a standard deviation of the error term taken from $\text{Var}[C(\mathbf{x}, \boldsymbol{\Theta})] = s^2$, ε is a random variable with zero mean and unit variance and $s\varepsilon$ is the error term of the model. The final form of the probabilistic model used to predict the flexural strength is as follows:

$$\sigma_c = \eta_D(x_D, \theta_D) \times \eta_H(x_H, \theta_H) \times \eta_M(x_M, \theta_M) \times \left[1/6\tau_i(AR)v_f^{2/3} + \sigma_m(1 - v_f^{2/3}) \right] + s\varepsilon \quad (6)$$

where $\eta_D(x_D, \theta_D) = \left[1 - \left(\frac{AR^{\theta_1} \times v_f - w^{\theta_3}}{UEI^{\theta_2} \times (SP/CNTS)^{\theta_4}} \right) \right]$, $\eta_H(x_H, \theta_H) = \left(\frac{t}{4+0.85t} \right)^{\kappa\theta_5}$, and $\eta_M(x_M, \theta_M) = \frac{(1+s/c)^{\theta_6}}{(w/c)^{\theta_7}}$ are critical relations after adding the unknown model parameters. To estimate the unknown model parameters, a Bayesian updating rule is used (Box and Tiao, 2011):

$$f(\boldsymbol{\Theta}) = \lambda L(\boldsymbol{\Theta})p(\boldsymbol{\Theta}) \quad (7)$$

where $\boldsymbol{\Theta}$ is a vector of unknown model parameters, $p(\boldsymbol{\Theta})$ is prior distribution of $\boldsymbol{\Theta}$ based on knowledge about $\boldsymbol{\Theta}$ that represents the information available before collecting the data, $L(\boldsymbol{\Theta})$ is the likelihood function that captures the information on $\boldsymbol{\Theta}$ from data, $\lambda = [\int L(\boldsymbol{\Theta})p(\boldsymbol{\Theta})d(\boldsymbol{\Theta})]^{-1}$ is a normalizing factor and $f(\boldsymbol{\Theta})$ is a posterior distribution. Once $f(\boldsymbol{\Theta})$ is known, the posterior mean vector and the covariance matrix are obtained. Because there is no prior information about $\boldsymbol{\Theta}$ before collecting data, a non-informative prior distribution $p(\boldsymbol{\Theta}) \propto \frac{1}{s}$ is used (Gardoni *et al.*, 2002). If new data becomes available in the future, Eq. (7) can still be used to update the estimates of the unknown model parameters by using the current $f(\boldsymbol{\Theta})$ as a new previous distribution. This approach has been extensively applied to various applications such as structural engineering and materials science (Choe *et al.*, 2007; Gardoni *et al.*, 2013; Gardoni *et al.*, 2009).

5 Probabilistic Model Assessment

A total of 105 published data was collected by the authors from several research papers to calibrate the unknown model parameters in Eq. (6) using a Bayesian methodology. The dataset is reported in (Ramezani, 2019c).

To evaluate the robustness of the proposed model, the total database is not used for the development of the probabilistic model due to two fundamental issues: overfitting and being overly optimistic about the model error. Therefore, the database is split into two sets of data using random selection; 80% of the data (84 data points) is used to develop the models (Dataset I) and the remaining 20% (21 data points) is used to validate the proposed probabilistic model (Dataset II). Table 2 shows the posterior statistics of $\boldsymbol{\Theta}$ estimated using Dataset I.

Table 2. Posterior statistics of the unknown model parameters using dataset I

Unknown Parameter	Mean	Standard Deviation	Correlation Matrix							
			θ_1	θ_2	θ_3	θ_4	θ_5	θ_6	θ_7	s
θ_1	0.393	0.0280	1	-0.539	0.998	0.950	0.861	-0.568	-0.977	0.628
θ_2	0.280	0.0086		1	-0.571	-0.697	-0.658	-0.040	0.608	-0.428
θ_3	0.481	0.0299			1	0.948	0.854	-0.567	-0.979	0.628
θ_4	0.132	0.0369				1	0.946	-0.388	-0.963	0.675
θ_5	1.059	0.0575					1	-0.213	-0.884	0.598
θ_6	0.173	0.0046						1	0.423	-0.529
θ_7	0.127	0.0146							1	-0.601
s	0.149	0.0008								1

The probabilistic model after estimating the unknown model parameters is shown below:

$$\sigma_c = \left[1 - \left(\frac{AR^{0.39} \times v_{f-w}^{0.48}}{UEI^{0.28} \times (SP/CNT_s)^{0.13}} \right) \right] \times \left(\frac{t}{4 + 0.85t} \right)^{1.06\kappa} \times \frac{(1 + s/c)^{0.17}}{(w/c)^{0.13}} \times \left[1/6\tau_i(AR)v_f^{2/3} + \sigma_m(1 - v_f^{2/3}) \right] \quad (8)$$

Figure 1 shows the performance of the proposed probabilistic model versus the experimental test data using Eq. (8). The 1:1 line (see the dashed line in Figure 1) indicates that the predicted flexural strength is identical to the experimental test data. The solid circles are the data used to develop the model (Dataset I) and the open triangles are the data used to validate the model (Dataset II). The robustness of the proposed probabilistic model is validated using Dataset II which is not used for model development, indicating the accuracy of the proposed probabilistic model by showing the desirable performance (i.e., within $\pm$ standard deviation of the error term in the proposed model; $s = 15\%$). Note that three data points inscribed within the oval in Figure 1 are related to the flexural strength of cement mortars reinforced with mechanical functionalized CNTs at different ages (Konsta-Gdoutos *et al.*, 2017). The mechanical functionalized CNTs provide superior interfacial bond strength, resulting in under-prediction of the flexural strength.

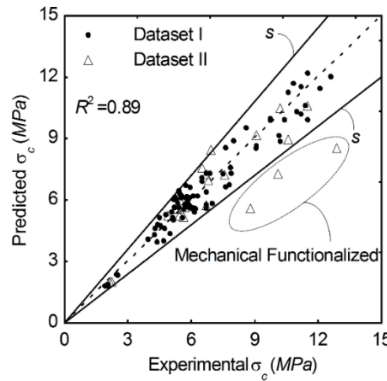


Figure 1. Predicted values vs. experimental test results for flexural strength.

Figure 2 shows the ratio of the predicted to the experimental value of the flexural strength ($R_F = \sigma_c^{pred.} / \sigma_c^{exp.}$; y-axis) versus different influential variables (x-axis; AR , v_f , κ , t , w/c ratio, s/c ratio, UEI , and SP/CNT_s ratio) when using the unknown model parameters shown in Table 1. The solid lines show $\pm 15\%$ of the model error. The mean and median values of R_F are 1.01 with the standard deviation of 0.11. In addition, R_F exhibits a normal distribution with respect to the reference line of $R_F = 1$ (i.e., the predicted flexural strength is identical to the experimental flexural strength), for all ranges of the investigated variables (x-axis). This suggests that the proposed critical relations can be reliably used to predict the flexural strength without any bias.

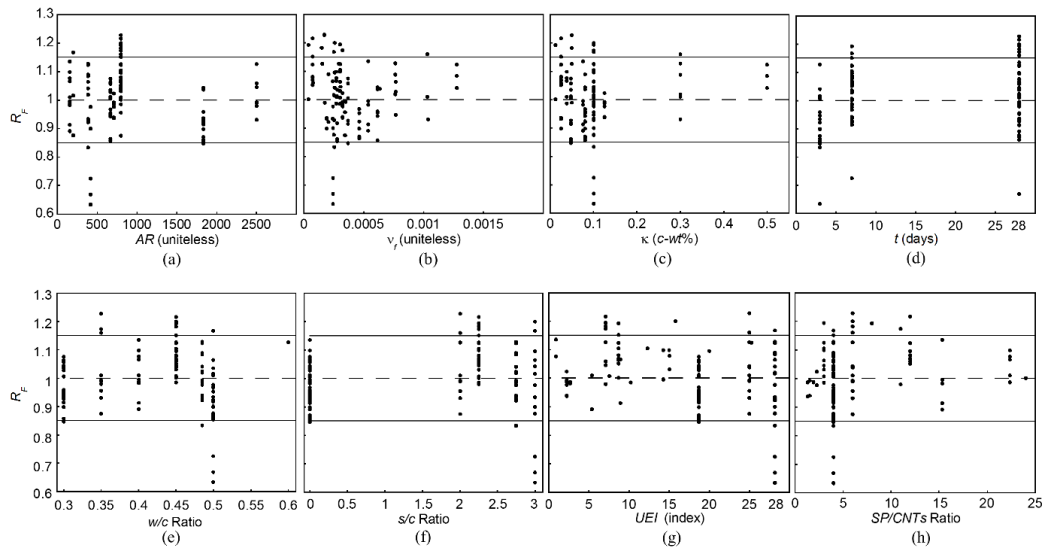


Figure 2. Influence of the experimental variables on R_F .

6 Conclusions

This paper proposes a bias-free probabilistic model based on the available deterministic Kelly-Tyson model to predict the flexural strength of CNT-cement nanocomposites (pastes and mortars). The proposed model considers the influences of CNT properties, dispersion state, matrix composition and cement hydration using a Bayesian approach. The proposed model is able to predict the flexural strength within $\pm 15\%$ error of the experimental test data. When additional data are available in the future, the proposed model can be updated with appropriate values or additional critical relations which can be discovered by other researchers.

Notations

σ_c : flexural strength of CNT-cement nanocomposite
 σ_m : flexural strength of the cement matrix without CNTs
 σ_f : tensile strength of the fiber
 v_f : CNT volume fraction
 d : CNT diameter
 L : CNT length
 τ_i : interfacial shear stress between CNTs and matrix
 AR : CNT aspect ratio
 w/c : water-to-cement ratio
 s/c : sand-to-cement ratio
 t : hydration age of cement
 $SP/CNTs$: superplasticizer-to-CNTs ratio
 UEI : ultrasonication energy indicator
 κ : CNT concentration
 η_D : dispersion relation
 η_H : hydration age relation
 η_M : matrix relation
 $C(\mathbf{x}, \boldsymbol{\Theta})$: probabilistic capacity model of interest (herein, σ_c)
 $\mathbf{x}$: set of measurable variables
 $C(\mathbf{x})$: deterministic model
 $\boldsymbol{\Theta}$: vector of unknown model parameters; $\boldsymbol{\Theta} = (\theta, s)$
 θ_i : set of unknown model parameters used to fit the data
 $\gamma(\mathbf{x}, \theta)$: correction term (herein, η_D , η_H and η_M)
 s : standard deviation of the error term
 ε : random variable with zero mean and unit variance
 se : error term of the model
 $p(\boldsymbol{\Theta})$: prior distribution of $\boldsymbol{\Theta}$
 $L(\boldsymbol{\Theta})$: likelihood function
 λ : normalizing factor; $\lambda = [\int L(\boldsymbol{\Theta})p(\boldsymbol{\Theta}) d(\boldsymbol{\Theta})]^{-1}$
 $f(\boldsymbol{\Theta})$: posterior distribution of $\boldsymbol{\Theta}$

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