

## Study on Strength and Gelation Time of Polyacrylamide/ Polyethyleneimine Composite Gels Reinforced with Coal Fly Ash for Water Shut-Off Treatment

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**ABSTRACT:** This study investigates the influence of coal fly ash (CFA) as a reinforcing material on the strength and the gelation of polyacrylamide (PAM)/polyethyleneimine (PEI) composite gels. Pure PAM/PEI gel and PAM/PEI gels containing CFA up to 2 wt % were synthesized via the cross-linking reaction between PAM and PEI solutions at room temperature (25°C) in distilled water dispersed with CFA. The strength of each composite gel was measured at temperature of 80°C, while the gelation was determined from 80°C to 95°C. Rheological measurements indicated that the strength of PAM/PEI composite gels filled with CFA contents was significantly rigid and stronger than that of pure PAM/PEI gel as a result of the enhanced interfacial interaction of well-dispersed CFA contents in PAM/PEI gel matrix. The gelation times of PAM/PEI gels containing CFA up to 2 wt % deviated from the gelation time of pure PAM/PEI gel. It was further observed that an increase in temperature resulted in a decrease in gelation time of PAM/PEI gel containing 2 wt % CFA. The scanning electron microscopy revealed the surface micrographs of PAM/PEI gels filled with CFA to be very dense without any noticeable micropores. The micropores were absent as scanning was performed on the dried composite gels. It also establishes the strong interaction between CFA and PAM/PEI gel matrix. Experimental findings showed that PAM/PEI composite gels reinforced with CFA are potential candidates for total water shut-off treatment in oilfields. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 2015, 132, 41392.

**KEYWORDS:** composites; cross-linking; gels; morphology; rheology

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### INTRODUCTION

Polymer gels are widely used during enhanced oil recovery to reduce excessive water production during oil exploration.<sup>1</sup> Water soluble polymers such as polyacrylamide (PAM) and polysaccharides have been used as means for improved oil recovery.<sup>2</sup> These polymers can be cross linked to form three-dimensional structures. In the case of PAM-based gels, the crosslinker can be either metallic or organic. Chromium is the most commonly used metallic crosslinker.<sup>3</sup> Unfortunately, the chromium crosslinkers, especially those based on Cr<sup>+6</sup> are known to be toxic and environmentally unacceptable. Another challenge with inorganic crosslinkers is their short gelation times at temperatures greater than 60°C.<sup>5,6</sup> In contrast, organically cross-linked gels possess good stability at elevated temperatures. This is attributed to the covalent bonds existing between the polymer and the organic crosslinker.<sup>7</sup> Several organically cross-linked gels have been reported in the literature.<sup>8</sup> These

gels are reported to be stable at temperatures up to 150°C,<sup>9</sup> but the use of phenol and formaldehyde as organic cross-linking agents has reduced drastically because they are not environmentally friendly.<sup>8</sup> Besides, polymer gels filled with solid particles have received great attention in recent times because of their excellent features which are not found in unreinforced polymer gels.<sup>10–14</sup> They possess distinct mechanical, optical, and electronic properties at a low loading of nanoscale fillers. Prajakta and Rajendra<sup>15</sup> reported an environmentally acceptable conformance gel system that incorporates nanosilica nanoparticles and an activator. Bani et al.<sup>16</sup> investigated in their study, the strength of different sandy PAM gel systems. Similarly, the potential of the clay-gel systems have been studied extensively.<sup>17–22</sup>

However, a comprehensive literature survey revealed that there is no scientific report to the best of our knowledge, on composite gels incorporated with coal fly ash (CFA). CFA is a useful

**Table I.** Properties of PAM and PEI Solutions

Variable	PAM	PEI
Molecular weight (g/mol)	250,000–500,000	70,000
Density <sup>a</sup> (g/cm <sup>3</sup> )	1.09	1.07
pH <sup>a</sup>	4	12

<sup>a</sup>Measured at room temperature (25°C).

by-product that can also be incorporated in polymer gels for various applications. Unlike previously reported solid particles incorporated in polymer gels, especially clay and carbon nanotubes, CFA is readily available, cheap, and has a significant impact on the gelation mechanism, strength, and morphological properties of composite polymeric gels developed in this study. Therefore, this article aims to investigate the strength and the gelation time of polyacrylamide/polyethyleneimine (PAM/PEI) composite gels reinforced with CFA. Detail explanations of the morphologies of these composite gels are also presented. Experimental results showed that these composite gel systems have great potentials in many fields, especially, for total water shut off in oilfields.

## EXPERIMENTAL

### Materials

PAM and PEI solutions used in this study were supplied by the SNF Chemical, France. The properties of PAM and PEI solutions are shown in Table I. The CFA used was purchased from the Saudi Ready-mix, Saudi Arabia. Distilled water containing 2 wt % NaCl<sup>23</sup> was used to prepare all the gelling solutions.

### Composite Gels Preparation

PAM/PEI composite gelling solutions containing 0.5 wt % (0.09 g), 1.0 wt % (0.19 g), and 2 wt % (0.38 g) CFA were prepared at room temperature (25°C). The composite gelling solutions filled with various CFA were developed by adding a measured quantity (in grams) of CFA into a 20 mL beaker containing a fixed volume of 10.39 mL distilled water followed by mixing using magnetic stirrer for 1 min. Then, fixed volumes of PAM and PEI solutions were added in drops (PEI solution was added last as crosslinker) and stirring was maintained for another 5 min. The volumes of PAM and PEI solutions were 4.29 and 0.32 mL, respectively. Also, pure PAM/PEI composite gel (without CFA) was prepared. The mix recipe and physical properties of each gelling solution are tabulated in Tables II and

**Table II.** Mix Recipe of Each Gelling Solution

Sample	CFA (g)	Distilled water (mL)	PAM (mL)	PEI (mL)
PAM/PEI (without CFA)	-	10.39	4.29	0.32
PAM/PEI + 0.5 wt % CFA	0.09	10.39	4.29	0.32
PAM/PEI + 1 wt % CFA	0.19	10.39	4.29	0.32
PAM/PEI + 2 wt % CFA	0.38	10.39	4.29	0.32

III, respectively. The procedure followed for the preparation of the composite gels was reported, elsewhere.<sup>24</sup> Chromium derivatives were used as cross-linking agents for partially hydrolyzed PAM in the preparation procedure followed.<sup>24</sup> However, in this study, we have chosen PEI as the cross-linking agent for PAM because it is environmentally benign and PAM-based gels cross-linked with PEI possess good stability at elevated temperatures.<sup>25</sup>

### Characterizations

The particle-size distributions of CFA were determined using Turbotrac S360 particle-size analyzer and the result is shown in Figure 1. It has a median size and an average particle size of  $d_{50} = 1.745$  and 24.58  $\mu\text{m}$ , respectively. X-ray diffraction (XRD) test of the CFA was conducted using Ultima IV model manufactured by Rigaku, Japan. The test was performed with scan speed of 2.5 deg/min within the range of 10–80 deg. According to XRD pattern of the CFA represented by Figure 2, the major minerals present are mullite ( $3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ ) and quartz ( $\text{SiO}_2$ ).<sup>26</sup> The JEOL scanning electron microscopy (SEM) was used to observe the morphology of the samples developed.

### Rheological Measurements

The gelling solutions were characterized using a TA discovery hybrid rheometer. Rheological experiments were conducted using 40 mm diameter parallel peltier plate geometry, a 1.00-mm gap, at a frequency of 1 Hz, and strain of 5%. This rheometer machine is equipped with a solvent trap system to prevent evaporation. The strength of pure PAM/PEI gel and PAM/PEI gels containing CFA contents was measured immediately after preparation without delay at temperature of 80°C unless otherwise stated.

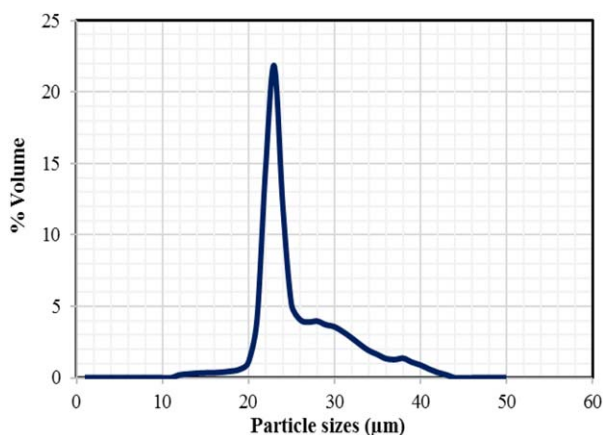
### Gelation Time Determination

Mortimer et al.<sup>27</sup> defines gelation time as the time required for the elastic and viscous moduli to intersect. Another definition for the gelation time is the time needed to reach specific gel strength.<sup>28,29</sup> Similarly, the point that corresponds to the maximum slope in the elastic modulus time curve has been defined as the gelation time.<sup>30</sup> In this study, the definition of gelation time adopted has been used by several authors,<sup>3,31</sup> which is defined as the time needed to reach the inflection point on the viscosity-time curve. The inflection point corresponds to the onset of the gel formation.<sup>32</sup> The gelation time of the composite gels developed was determined in the temperature range of 80°C–95°C. Each sample was measured using a TA discovery hybrid rheometer. The gelation test of each sample was performed with a parallel plate and peltier plate geometry. To avoid the evaporation of gelling solutions during measurements, the

**Table III.** pH and Density of Each Gelling Solution

Sample	pH <sup>a</sup>	Density <sup>a</sup> (g/cm <sup>3</sup> )
PAM/PEI (without CFA)	10.55	1.002
PAM/PEI + 0.5 wt % CFA	10.58	1.004
PAM/PEI + 1 wt % CFA	10.70	1.007
PAM/PEI + 2 wt % CFA	10.88	1.014

<sup>a</sup>Measured at room temperature (25°C).



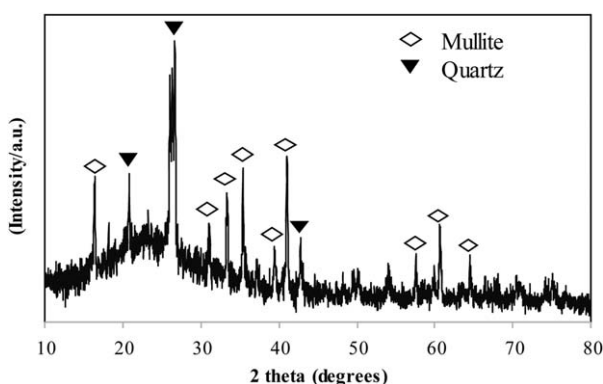
**Figure 1.** The particle-size distributions of CFA,  $d_{50} = 1.745$ . [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

gap between the two plates was covered with the solvent trap system.

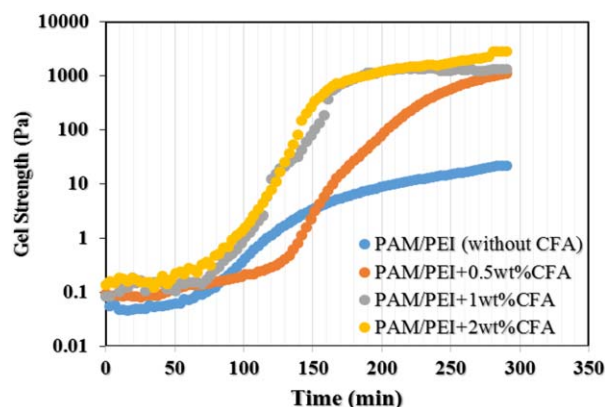
## RESULTS AND DISCUSSION

### Strength of the PAM/PEI Composite Gels Containing CFA

To investigate the influence of CFA incorporation on the strength of pure PAM/PEI gel, PAM/PEI gels containing various CFA contents (i.e., 0.5, 1, and 2 wt %) were prepared. Figure 3 shows the gel strength curves as a function of reaction time for pure PAM/PEI gel and PAM/PEI gels containing CFA up to 2 wt %. It can be seen that the strength of pure PAM/PEI gel rise steadily and reached a plateau at 270 min of the reaction time. Its strength was 21 Pa. However, on addition of 0.5 wt % CFA to the pure PAM/PEI gel, a sharp increase in the strength was noticed. Its strength increased considerably from 21 to 1099 Pa. Likewise, PAM/PEI gels containing 1 wt % CFA and 2 wt % CFA, respectively, enhanced the strength of pure PAM/PEI gel significantly with PAM/PEI gel containing 2 wt % CFA having the highest strength. The strength of CFA-free gel (i.e., pure PAM/PEI gel) and PAM/PEI gel containing 2 wt % of CFA was 21 and 2814 Pa, respectively. This implied that the strength of PAM/PEI gel filled with 2 wt % CFA was 134-fold greater than that of the pure PAM/PEI gel as a result of the enhanced interfacial interaction of well-dispersed CFA contents in PAM/PEI gel matrix. Thus, this observation suggests that PAM/PEI gels



**Figure 2.** XRD pattern of the CFA used in this study.<sup>26</sup>

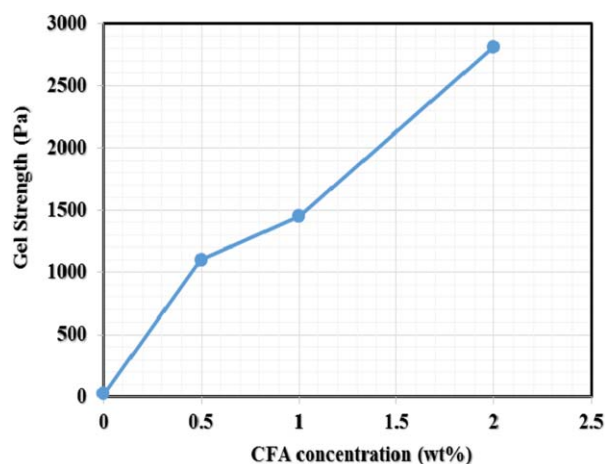


**Figure 3.** Gel strength curves for pure PAM/PEI gel and PAM/PEI gels containing CFA up to 2 wt %. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

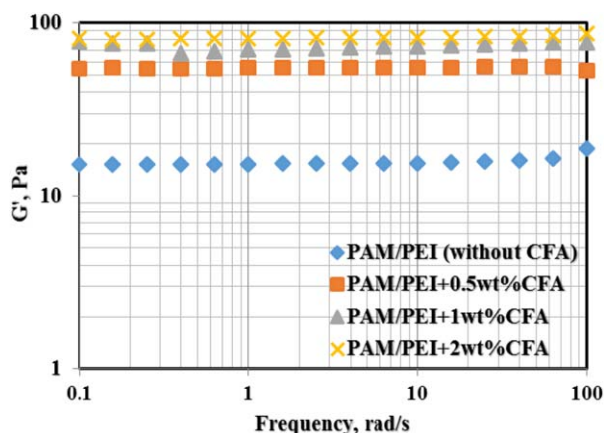
reinforced with CFA were substantially rigid and stronger than pure PAM/PEI gel. Figure 4 elucidates the strength of each composite gel as a function of CFA concentration (wt %). It could be noticed that the gel strength increases with the increase in CFA content.

It is noteworthy to point out that the strength values of PAM/PEI composite gels containing CFA in this study are in agreement with previous work on PAM-based gels reinforced with solid particles.<sup>33–35</sup> However, the major advantage of the present composite gels over many PAM-based gels, especially PAM-based gels crosslinked with inorganic crosslinkers, lies in the fact that, PEI (an organic crosslinker) forms thermally stable gels with PAM-based gels and its derivative.<sup>25,31,36,37</sup> Therefore, PAM/PEI gels filled with CFA are economical gel systems, which combine excellent compatibility (both gel matrix and CFA filler) with high strength values. These composite gels can be utilized for water shut-off treatment in oilfields, especially in formations with large open channels.

To further characterize the strength of these composite gels, frequency sweep mode was performed. Oscillatory frequency

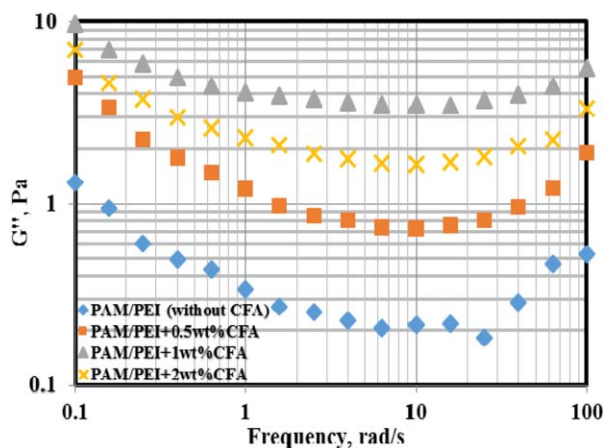


**Figure 4.** Gel strength as a function of CFA concentration. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 5.** Elastic modulus of pure PAM/PEI gel and PAM/PEI gels containing CFA up to 2 wt %. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

sweep tests were conducted after 1 month of keeping the gels at room temperature. In this case, the gels have become rigid. Figure 5 displays the elastic modulus,  $G'$ , measured in the frequency range  $\omega = 0.1$ –100 rad/s for the pure PAM/PEI gel and PAM/PEI gels filled with CFA up to 2 wt %. It is observed that  $G'$  demonstrated very weak dependency on  $\omega$  over the range 0.1–10 rad/s and reached a plateau modulus at  $\omega < 1$  rad/s. This observation confirms the formation of a gel network of rubbery nature. A similar observation was previously reported for polymer gels.<sup>38–40</sup> These results showed that  $G'$  of PAM/PEI gels containing CFA was higher than that of pure PAM/PEI gel throughout the whole frequency range, indicating that PAM/PEI gels filled with CFA were strong and rigid. The viscous modulus,  $G''$ , of each gel is shown in Figure 6. It is obvious that  $G''$  of virgin PAM/PEI gel and PAM/PEI containing CFA contents decreases in the frequency range  $\omega = 0.1$ –10 rad/s and increases afterward. In general, as  $G'$  is practically higher than  $G''$  in each composite gel, it implies that solid-like behavior is predominant in all these composite gels with PAM/PEI gel filled with 2 wt % CFA demonstrating highest solid-like character.



**Figure 6.** Viscous modulus of pure PAM/PEI gel and PAM/PEI gels containing CFA up to 2 wt %. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

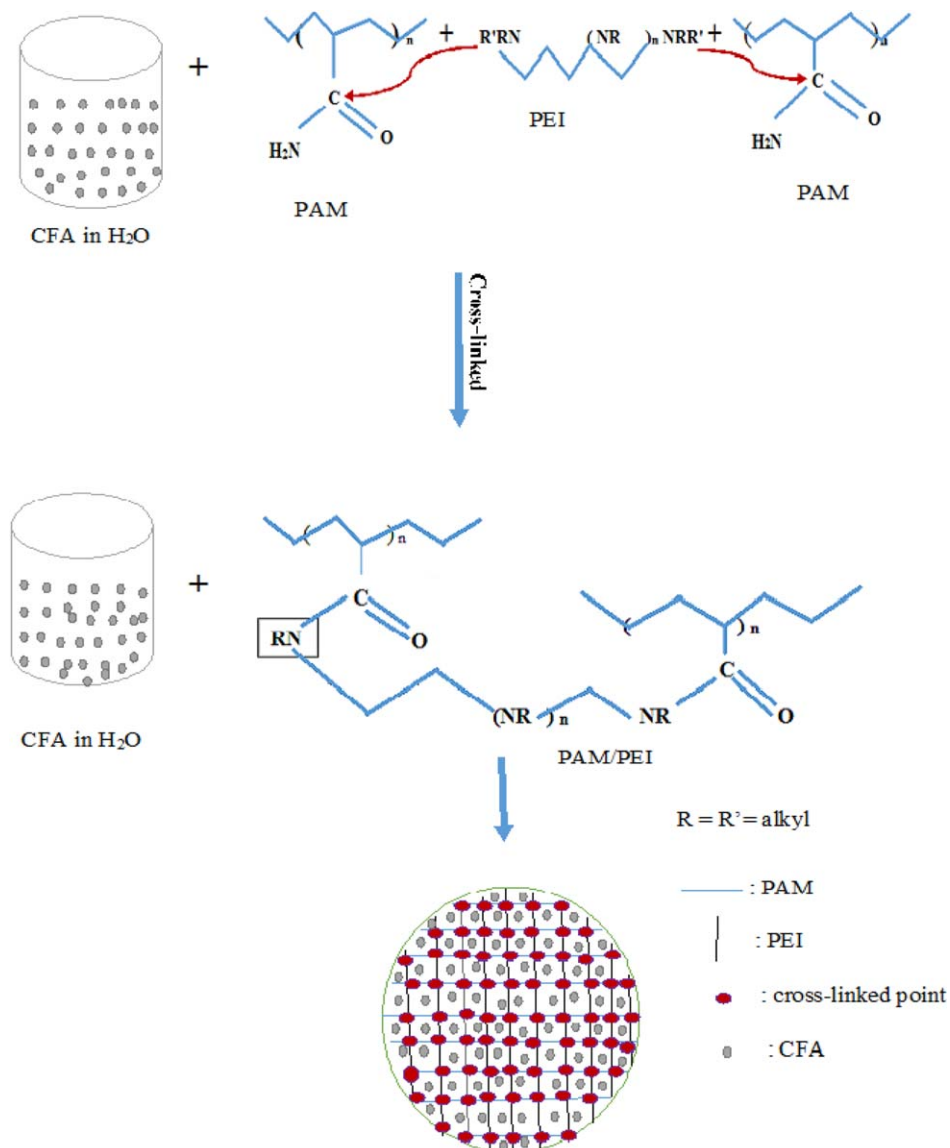
### Gelation Mechanism

Al-Muntasheri et al.<sup>41</sup> and Reddy et al.<sup>42</sup> have discussed extensively in their studies, the gelation mechanism of PAM cross-linked with PEI. It is believed that the gelation process of PAM/PEI gel occurred through transamidation mechanism. This reaction is a substitution reaction where the nucleophilic amine nitrogen on PEI substitutes the acrylamide pendant groups on the PAM chain which would result into a covalent bond between the nitrogen on PEI and the PAM amide group. Research studies have shown that variety of polymers containing acrylamide pendant groups react with PEI nitrogen via a transamidation reaction pathway to produce gels.<sup>42</sup> Incorporation of CFA to the PAM/PEI gel is expected to enhance its strength in one hand, and also produce suitable gelation time on the other. Figure 7 demonstrates a typical schematic gelation mechanism of crosslinked PAM/PEI composite gel containing CFA dispersed in distilled water.

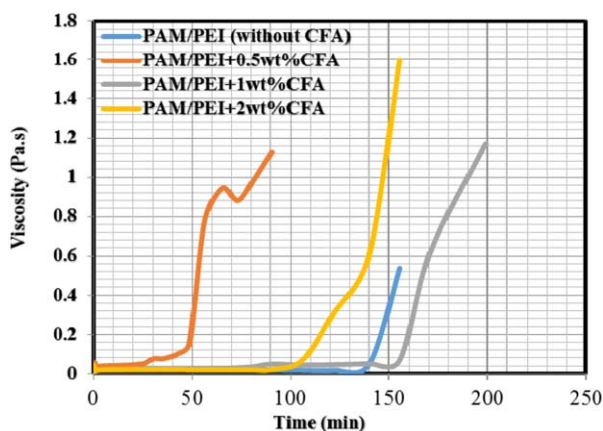
### Gelation Time Comparison Between Pure PAM/PEI Gel and PAM/PEI Gels Containing CFA

Gelation time is one of the important features of polymeric gel systems. It is, therefore, necessary to compare the gelation times of pure PAM/PEI gel and PAM/PEI gels containing various CFA contents. Figure 8 represents the viscosity evolution as a function of time for the pristine PAM/PEI gel and PAM/PEI gels containing CFA ranging from 0.5 to 2 wt %. It can be observed that the gelation of pure PAM/PEI gel started at 139 min while the gelation kinetics of PAM/PEI gel containing 0.5 wt % CFA began after 30 min and proceed much faster than the gelation time of pure PAM/PEI gel. This observation suggests that the addition of CFA (0.5 wt %) opens the cross-linking sites more rapidly, thereby allowing the PEI to crosslink very fast with PAM. In contrast, the gelation times of PAM/PEI composite gels filled with 1 and 2 wt % CFA were 153 and 103 min, respectively, which are longer than the gelation time of PAM/PEI gel filled with 0.5 wt % CFA. The possible explanation for this behavior could be that the incorporation of 1 and 2 wt % CFA made the cross-linking sites unavailable for PEI and PAM to react on time, and this resulted in the delay of gelation time. In all cases, it is observed that the gelation times of PAM/PEI gels containing CFA up to 2 wt % deviated from the gelation time of pure PAM/PEI gel.

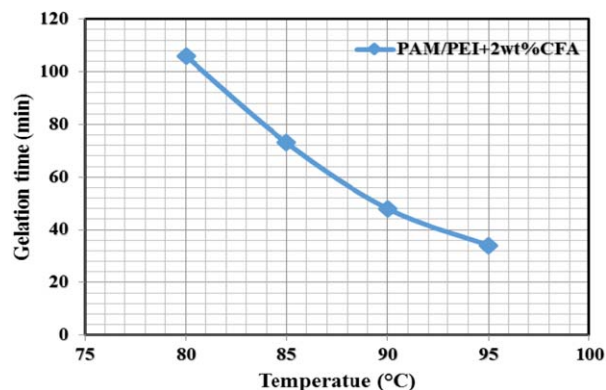
Several repeated tests in our laboratory on the gelation kinetics of PAM/PEI gels filled with CFA up to 2 wt % produced similar results. The possible conclusion that could be drawn from these observations is that, there is high tendency of the disproportionate dissolution of CFA ions emanating from quartz and mullite (as observed in XRD pattern) as well as the different particle sizes of CFA within the composite gels might have contributed to the gelation time variations. The gelation of PAM/PEI gels filled with various CFA contents could better be understood by carrying out thermal analysis on the gel systems using the differential scanning calorimetry (DSC). The dynamic DSC study will reveal peaks at various temperatures in each gel and these could be correlated with available models (e.g., Avrami model). Also, the dynamics light scattering and the small neutron scattering instruments could be used to confirm unavailability of the cross-linking sites, investigate the composite gels



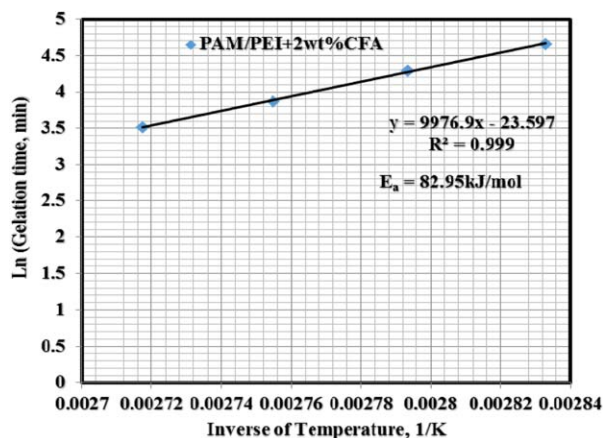
**Figure 7.** A typical gelation mechanism of PAM/PEI composite gel containing CFA. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 8.** Viscosity evolution of pure PAM/PEI gel and PAM/PEI gels containing CFA up to 2 wt %. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



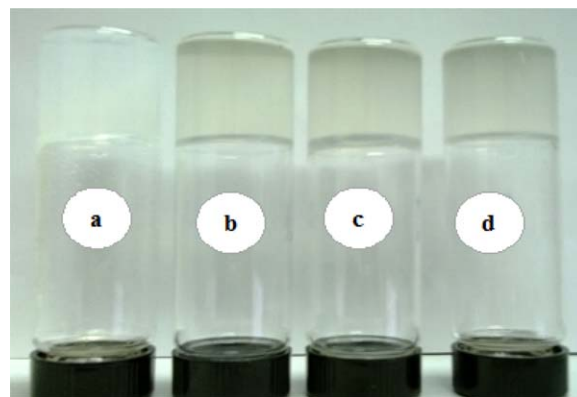
**Figure 9.** Effect of various temperatures on the gelation time of PAM/PEI gel containing 2 wt % CFA. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 10.** Arrhenius plot of gelation time and various temperatures for the PAM/PEI gel containing 2 wt % CFA. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

network structures, and the way CFA interact within PAM/PEI matrix. Further studies are, therefore, recommended to investigate the network structures and gelation mechanism of these systems using the aforementioned instruments.

Nevertheless, the gelation time of the composite gels can still be controlled by modifying PAM/PEI formulation ratios<sup>41</sup> and by varying the quantity of CFA in the PAM/PEI formulations. However, high concentration of PEI in reinforced PAM/PEI

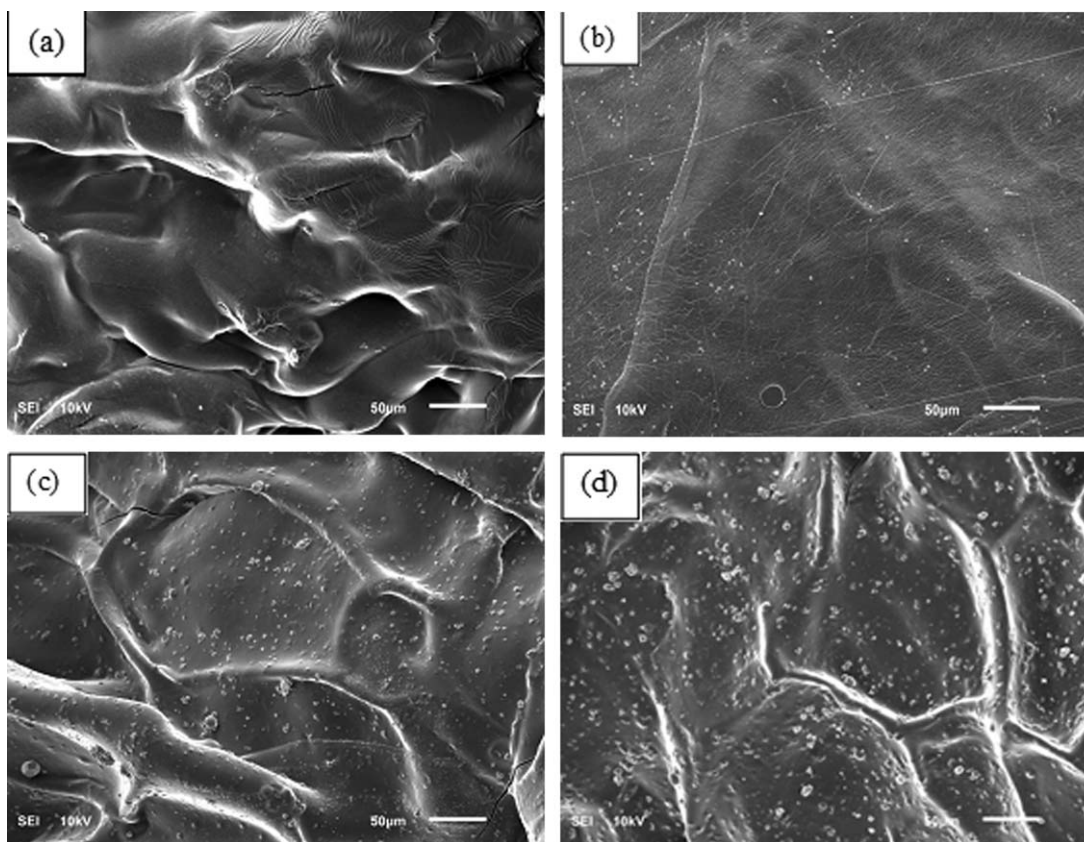


**Figure 11.** Nonflowing gels: (a) PAM/PEI (without CFA); (b) PAM/PEI + 0.5 wt % CFA; (c) PAM/PEI + 1 wt % CFA; and (d) PAM/PEI + 2 wt % CFA. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

composite gels should be avoided in order not to lead to syneresis (reduction in gel volume as a result of water expulsion).<sup>43</sup>

#### Effects of Various Temperatures on the Gelation of PAM/PEI Gel Containing CFA

Temperature is one of the key parameters that determines the gelation time. Therefore, the effects of various temperatures on the gelation time of PAM/PEI gel containing 2 wt % CFA were investigated. The temperature ranges from 80°C to 95°C with



**Figure 12.** SEM image of each composite gel: (a) PAM/PEI (without CFA); (b) PAM/PEI + 0.5 wt % CFA; (c) PAM/PEI + 1 wt % CFA; and (d) PAM/PEI + 2 wt % CFA.

5°C increment. Focusing on Figure 9, it is noticed that an increase in temperature results in a decrease in gelation time of PAM/PEI gel containing 2 wt % CFA. The decrease in gelation time as temperature increases is an indication of endothermic type of reaction.<sup>44</sup> Moreover, with the increase in temperature, the degree of hydrolysis of PAM increases, which in turn increases the number of cross-linking sites.<sup>41,45</sup>

Furthermore, the gelation time of this composite gel was correlated to temperature according to the Arrhenius type eq. (1):

$$GT = M \exp\left(\frac{E_a}{RT}\right) \quad (1)$$

where  $GT$  is the gelation time in minutes,  $M$  is the frequency factors in minutes,  $E_a$  is the activation energy in kJ/mol,  $R$  is the universal gas constant in kJ/mol K and  $T$  is the absolute temperature in K.<sup>3</sup>

From eq. (1), a plot of the natural log of  $GT$  versus  $1/T$  generates a straight line with a slope of  $E_a/R$  and an intercept of  $\ln M$ . Figure 10 represents the Arrhenius relation between gelation time and temperatures for the PAM/PEI gel filled with 2 wt % CFA. The activation energy of this composite gel was measured to be 82.95 kJ/mol. This value is consistent with the values obtained by Jurinak et al.<sup>46</sup> Interestingly, the activation energy of this composite gel indicates that its gelation can form slowly and can, therefore, be utilized in applications where very intense gel placement is required.

#### Morphologies of Pure PAM/PEI Gel and PAM/PEI Gels Containing CFA

Figure 11(a–d) shows the images of the nonflowing PAM/PEI composite gel and PAM/PEI composite gels containing CFA up to 2 wt %. These gels were dried and their surface morphologies were examined by the SEM. The micrographs of pure PAM/PEI gel and PAM/PEI gels filled with CFA contents are depicted in Figure 12(a–d). It can be observed from the surface morphology of pure PAM/PEI gel [Figure 12(a)] that its surface has “hole-like” structure in some regions which probably makes it less dense and contributes to its low strength. In contrast, the surface morphologies [Figure 12(b–d)] of PAM/PEI gels filled with CFA up to 2 wt % exhibited very dense surfaces in almost every region which probably enhanced their high strength values. Also, it can be noticed that the surface micrograph of PAM/PEI gel containing 0.5 wt % CFA was different from that of PAM/PEI gels filled with 1 and 2 wt %, respectively. The reason for the observed differences in the morphologies of the gels could be attributed to the influence of CFA compositions which probably alter the surface morphologies of the composite gels. Further, it is expected to have observed very dense compact surface structures in the gels filled with CFA contents as scanning was performed using dried gels. Previous studies on composite gels have reported similar observations.<sup>22,47,48</sup> Besides, it is evident from the surface morphologies of PAM/PEI composite gels filled with CFA that CFA is well dispersed without agglomeration. This observation supported the fact of strong interaction between PAM/PEI gel and CFA filler.

#### CONCLUSIONS

In this study, the strength and the gelation time of cross-linked polyacrylamide/polyethyleneimine (PAM/PEI) composite gel

containing various contents of coal fly ash (CFA) were investigated using rheological approach. PAM/PEI composite gels filled with CFA were developed via the cross-linking reaction between PAM and PEI solutions in distilled water containing various CFA contents. Incorporation of CFA has a pronounced influence on the strength and the gelation of pure PAM/PEI gel. Experimental findings showed that the strength of PAM/PEI composite gels containing CFA contents was substantially rigid and stronger than that of pure PAM/PEI composite gel. The strength of PAM/PEI gel reinforced with 2 wt % CFA was 134-fold greater than that of pure PAM/PEI gel. The gelation times of PAM/PEI gels containing CFA up to 2 wt % were found to deviate from the gelation time of pristine PAM/PEI gel. Moreover, the gelation time of PAM/PEI gel filled with 2 wt % CFA decreased with the increase in temperature due to the hydrolysis of PAM as temperature increases. The SEM revealed the surface morphologies of PAM/PEI gels reinforced with CFA contents to be very dense without any noticeable micropores. The micropores were absent as scanning was performed on the dried composite gels. SEM characterization also confirms the strong interaction between CFA and PAM/PEI gel matrix.

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