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A Study on Effect of Solved Caffeine on Curing and Thermal Degradation Behavior of Cyanoacrylate

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Abstract
The aim of this study is to investigate the thermal behavior of cyanoacrylate in presence of solved caffeine. For this purpose, DSC and TGA tests were used. Results show that by increasing in amount of caffeine, decrease in the released energy of curing, initiation time and duration of polymerization will occur. It was also shown that presence of caffeine results in increase in degradation temperature of cyanoacrylate mixture in comparison to pure cyanoacrylate.

Keywords: Cyanoacrylate-Caffeine-Thermal behavior-DSC-TGA

Introduction
Nowadays, solvent-free, one-part cyanoacrylate adhesive widely used in a range of industries including the automotive, beauty aid, electrical, electronic, machinery, medical, and plumbing [1-3]. According to literature survey done by the authors, there are few papers concentrated on thermal behavior of cyanoacrylate in presence of other particles as modifiers. Hence, the main goal of current research has been focused on clarifying the role of solved caffeine in cyanoacrylate.

Experimental
In order to prepare samples, 1wt% of para-toluane sulfunic acid was solved in cyanoacrylate. Then five different amount of caffeine according to Table 1, were solved in the mixture. All of these steps were done by hand. Isothermal Differential Scanning Calorimetry (Isothermal DSC) was used to investigate energy release, initiation time of polymerization and duration of polymerization. For this purpose, sufficient amount of mixture was placed in the DSC device in room temperature and curves were obtained. In order to acheive degradation temperature of the polymer, Thermogravimetric Analysis (TGA) was utilized. For this purpose, cured polymer was heated up to 500°C by the rate 10°C/min.

Results and Discussion
Results of isothermal DSC are shown in Fig. 1 and Table 1. It can be observed that by increasing the caffeine to acid ratio, the energy released during polymerization, the time of initiation of polymerization and duration of polymerization, has decreased. The cyanoacrylate monomers can be polymerized by both free radical and anionic mechanisms, but the latter hold much greater interest. This anionic reaction is inhibited at pH < 5.5, hence monomers are kept in their liquid form by the addition of a weak acid during storage and prior to use [4]. Prior to polymerization, neutralization of the acids takes place. Any practical, weak electron donor base, such as moisture on the surface of a substrate, can trigger the polymerization reaction. Basic chemicals such as sodium carbonate, phosphates, amines, caffeine or pyridine can be used to neutralize the stabilizer and so accelerate polymerization [1,5]. More amount of caffeine (as an electron donor), decrease the initiation time of polymerization and also the time that is needed for full polymerization. The decrease in energy that is released during polymerization is because of the fact that more link formation will be prevented by caffeine. By increasing the caffeine to acid ratio, link formation will be more hindered and hence the released energy decreases.

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TGA results are shown in Fig. 2 and Table 2. It can be observed that there is an increase in temperature of 10% and 90% degradation of polymer in presence of caffeine in comparison with pure cyanoacrylate. By increasing the temperature, caffeine absorb heat in order to degradation thus, the degradation temperature will increase in presence of caffeine. It can also be obtained from the TGA curves that increase in caffeine to acid ratio results in decrease of percentage of degradation. As it was mentioned before, this happens because of the heat that is absorbed by caffeine during degradation.

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Table 1. Results Obtained from DSC tests.

Caffeine to acid ratio	Time of initiation (min)	Duration of polymrization (min)	Energy released (J/g)
1.5	163	137	202.64
6	157	123	190.43
9	152	118	173.59
12	125	110	165.66
15	120	105	149.11

Table 2. Results Obtained from TGA tests.

Caffeine to acid ratio	10% degradation temp. (°C)	90% degradation temp. (°C)
0	181.27	252.72
1.5	218.54	248.92
6	219.21	248.08
9	218.37	258.96
12	222.04	258.31
15	218.37	287.97

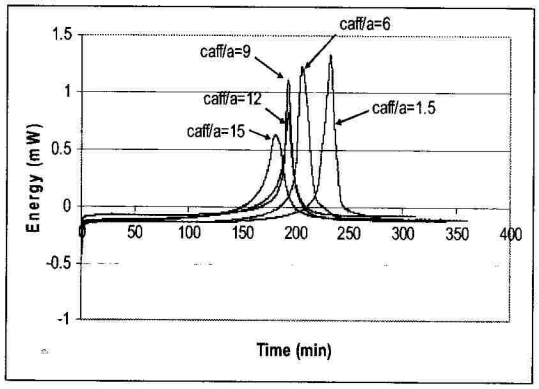

Fig 1. Curves obtained from DSC tests.

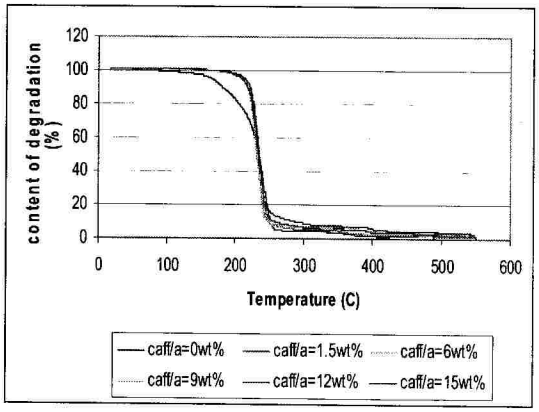

Fig 2. Curves obtained from TGA tests.