# A novel preparation of polyaniline in presence electric and magnetic fields

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**Abstract.**  We have described primary studies on conductivity and molecular weight of polyaniline separately in the electric and magnetic fields when it is used in a field effect experimental configuration. We report further studies on doped in-situ deposited polyaniline. First we have chemically synthesized polyaniline by ammonium peroxodisulfate in acidic aques and organic solutions at different times. Then we measured mass and conductivity and obtained the best time of polymerizations. In continue, we repeated these reactions separately under different electric and magnetic fields in constant time and measured mass and conductivity. The polyaniline is characterized by gel permeation chromatography (GPC), UV-Visible spectroscopy and electrical conductivity. High molecular weight polyanilines are synthesized under electric field, $M_w$ = 520000-680000 g/mol, with $M_w/M_n$ = 2-2.5. The UV-Visible spectra of polyanilines oxidized by ammonium peroxodisulfate and protonated with dodecylbenzenesulfonic acid (PANi-DBSA), in N-methylpyrolidone (NMP), show a smeared polaron peak shifted into the visible. Electrical conductivity of polyanilines has been studied by four-probe method. The conductivity of the films of emeraldine protonated by DBSA cast from NMP are higher than 500 and 25 S/cm under 10 KV/m of potential) electric field and 0.1 T magnetic field, respectively. It shows an enhanced resistance to ageing too. By the next steps, we carried chemical polymerization at the best electric and magnetic fields at different times. Finally, resulted in finding the best time and amount of the fields. The longer polymerization time and the higher magnetic field can lead to degradation of polyaniline films and decrease conductivity and molecular mass.

**Keywords:**    chemical polymerization; polyaniline; electric field; magnetic field

## 1. Introduction

   Since then it has been discovered that polyaniline (PANi) is the generic polymer of a novel class of conducting polymers (MacDiarmid *et al*. 1984), it has become the object of numerous studies. A synthesis derived from Green's synthesis that becomes a standard procedure. This standard PANi in the form of EB is only partly soluble in 1 - methyl - 2- pyrrolidinone (NMP) (Liao *et al*. 1995 and Hosseini *et al*. 2005). The soluble part has a low molecular weight (Angelopoulos *et al*. 1996) (MW): $M_n$ = 20000 - 26000 g/mol, $M_w$ = 30000 - 60000 g/mol, a large

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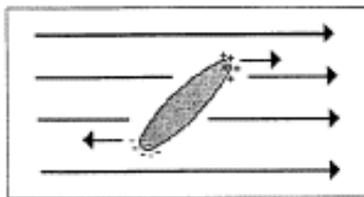∗Corresponding author, Ph.D., E-mail: davidtyona@yahoo.com

Fig. 1 Orientation of a polar molecule in electric field

polydispersity $M_w/M_n$ = 2.5 - 3 and a low inherent viscosity, $\eta_{inhb}$ = 0.8 -1.2 dl/g.

Polymers that can move charge carriers can be classified in two groups. The first one has ionic carriers while the other group has electronic carriers. Conjugated polymers such as polypyrrole, polyaniline and their derivatives can be classified between these two groups according to the temperature and humidity environmental conditions. Therefore, electric field effects of selected doped on conducting polymers were investigated (MacDiarmid *et al*. 2001). Fig. 1 shows orientation of polar oxidative form aniline in electric field. The polar oxidative aniline is rotating toward positive and negative electric field.

The magnetic field can influence on the chemical reaction that containing radicals and induce orientation of most organic polymer molecules and biological macromolecules (Sugiyama *et al*. 1992). Some articles concerning the effect of the magnetic field on the electrochemistry have been published (Wan and yang 1995), they found that electropolymerization rate of PANi could clearly be enhanced under a magnetic field. Electrochemical synthesis of polyaniline nanoparticles in the presence of magnetic field and erbium chloride have been reported by Rongguan L. (Rongguan *et al*. 2005).

Orientation techniques commonly used on conducting polymers to improve their conductivity and or to induce anisotropy of conductivity. The orientation of the polymer chains can be obtained by polymerization in an oriented nematic solvent. This carried out by mechanically stretching the polymer or by deposition on surfaces. By these processes, the orientation occurs via an external constraint and therefore displays a non reversible behaviour. We reported the electrical field influence on molecular mass and electrical conductivity of poly (2 - ethanolaniline) under electric field (Hosseini *et al*. 2013). In this paper, we have described preliminary studies on the dependency of the conductivity of PANi to using electric and magnetic fields when used in a field effect experimental configuration. Now we report further studies on different conditions and also studies on doped in-situ deposited polyaniline.

## 2. Experimental

### 2.1 Reagents and instrumentation

Chemicals used in this study were American Chemical Society (ACS) grade. Aniline (Merck) was dried with NaOH, fractionally distillated under reduced pressure from sodium or $CaH_2$. Other chemicals and solvents were purified as pre-standard procedure before use.

Conductivity changes were measured with a four probe device (home made, ASTM Standards, F 43-93). Electric field device was applied by Hipotronics S.O. No. 004390-00, HV power supply,

model 830.50 made in USA. Magnetic field device was applied by Moliantoss. Co. HV power supply.AC. 0-5 KV. model 5115,1092 made in Iran. A fourier-transform infrared spectrometer, FT-IR, (Bruker) was used in spectral measurements of the polymer and graft copolymer and reported (sh = sharp, w = weak, m = medium, b = broad). UV-Vis spectra were recorded using a Perkin Elmer Lambda 15 spectrophotometer on 0.05 wt./vol. EB solutions in spectrophotometric grade NMP. Molecular weight was measured at $30^o$C with a gel permeation chromatography (GPC), (Waters Associates, model 150 - C). Three styragel packed columns with different pore sizes ($10^4$-$10^6$$A^0$) were used. The mobile phase was NMP with flow rate of 1.5 ml/min.

## 2.2 Preparation of polyaniline in the absence of electric field

Project participants followed the same instructions to oxidize 0.001 mol aniline hydrochloride with 0.001 mol ammonium peroxydisulfate, $(NH_4)_2S_2O_8$, in aqueous medium. Aniline hydrochloride (purum; 0.1 g) was dissolved in 10 ml HCl 1 M in a 50 ml volumetric flask. Ammonium peroxydisulfate (purum; 0.228 g) was dissolved in 10 ml HCl 1 M, then was added. Both solutions were kept for different times (5, 10, 15, 20, 30, 40, 50, 60, 120 min) at room temperature (~18-24℃). The PANi precipitate was collected on a filter, washed with three 10-mL portions of 0.1 M HCl, and acetone. Polyaniline hydrochloride powders were dried in air and then in vacuum at 60℃. Then its weights and electrical conductivities were measured by four probe method (Hosseini *et al*. 2005 and 2003).

PANi powder with $(NH_4)_2Ce(NO_3)_6$, $FeCl_3$ (dry), $FeCl_3.6H_2O$, $Fe(ClO_4)_3$ and $H_2O_2$ as initiators were synthesized in similar manner. Therefore, PANi powder were prepared in similar manner with $H_2O$, ethanol, tetrahydrofuran (THF), m-crosol, dimethylsulfoxide (DMSO), acetone, acetonitrile, cyclohexane, dichloromethane, benzene and nitrobenzene as solvents. These solvents have different dielectric constants.

## 2.3 Preparation of polyaniline under electric field

Polyaniline was prepared under the same chemical circumstance but with different electric fields in 45 min at room temperature. Therefore, polyanilines prepared by the same reaction but various time spans at the best electric field. Then its weights and electrical conductivities were measured.

## 2.4 Preparation of polyaniline in presence of magnetic field

Project participants followed the same instructions to oxidize 0.0005 mol aniline hydrochloride with 0.0005 mol ammonium peroxydisulfate in aqueous medium. Aniline hydrochloride (purum; 0.05 g) was dissolved in 1 ml HCl 1 M in a test tube. Ammonium peroxydisulfate (purum; 0.114 g) was dissolved in 4 mL HCl 1 M, then was added. Both solutions were kept for different times at room temperature (~18-24℃). The PANi precipitate was collected on a filter, washed with three 10-mL portions of 0.2 M HCl, and similarly with acetone. Polyaniline hydrochloride powder was dried in air and then in vacuum at 60℃.

Therefore, polyanilines prepared by same reaction under different magnetic fields at the best time. Additional polymerizations were carried out in different organic phases and dopants in absence and under magnetic field. Then its weights and electrical conductivities were measured.
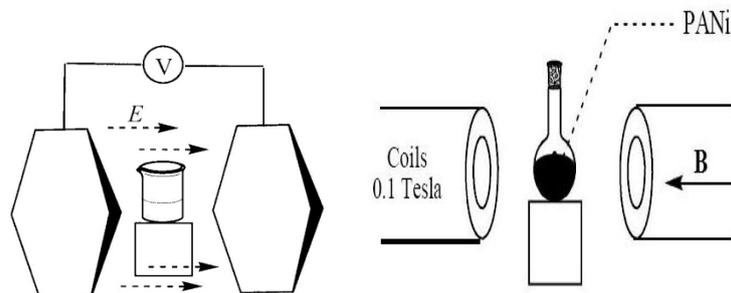
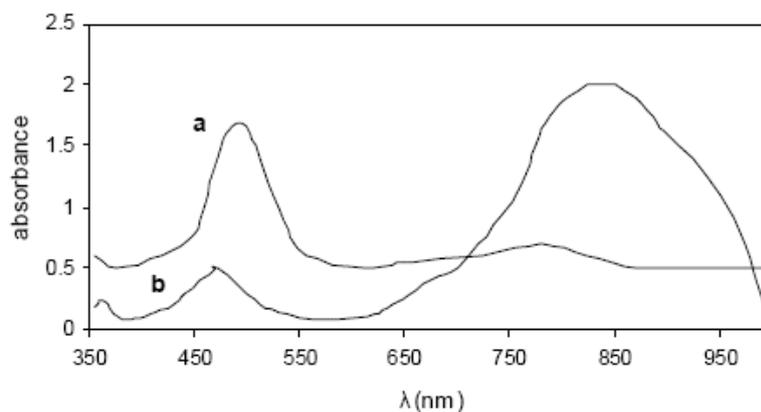Fig. 2 The polymerization cell of (a) electric and (b) magnetic Fields



Fig. 3 UV-visible of PANi prepared (a) without and (b) in presence electric field in NMP

## 3. Results and dsicussion

The polymer which was obtained is of high purity because the excess initiator was readily washed with acetone and the purity was confirmed by elemental analysis. Conductivity of all samples measured at room temperature by a four-probe method on pellets compressed at 700 MPa, 13 mm in diameter and 1-1.5 mm thick (Hosseini *et al*. 2006 and Hosseini 2006). Fig. 2 shows devices of a) electric and b) magnetic fields used for this experiment.

Fig. 3 shows UV-visible spectroscopy for PANi oxidized by ammonium peroxydisulfate and protonated with DBSA that prepared in (a) without and (b) with presence of electric field in NMP. UV-Visible spectrum of PANi prepared in presence of electric field shows a peak in higher than 650-950 nm. It is related to $\pi$ - $\pi^*$ transitions, high and longer than molecular chain.

Tables 1 and 2 show electrical conductivity and weight of polyaniline produced in the absence of any field in different times and initiators, respectively. Electrical conductivity and weight of PANi increases by the increase in duration of polymerization. Ammonium peroxydisulfate and ammonium ceric nitrate of oxidants are better than other oxidants.

Tables 3 and 4 showed electrical conductivity of polyaniline produced by ammonium

Table 1 Electrical conductivity of polyaniline produced by $(NH_4)_2S_2O_8$ (HCl 0.1M) as initiator in different times and without electric field

| Sample | Time of polymerization (min) | Color change time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|--------|------|------|------|--------|
| 1 | 10 | 3 | 0.07 | 0.0005 |
| 2 | 15 | 3 | 0.07 | 0.01 |
| 3 | 20 | 3 | 0.09 | 0.8 |
| 4 | 30 | 3 | 0.15 | 7 |
| 5 | 40 | 3 | 0.17 | 8 |
| 6 | 60 | 3 | 0.20 | 8 |
| 7 | 120 | 3 | 0.22 | 10 |

Table 2 Electrical conductivity of polyaniline produced by different initiators in water and without electric field after 45 min

| Initiator | Color change time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|--------|------|------|--------|
| $(NH_4)_2S_2O_8$ | 3 | 0.19 | 0.08 |
| $(NH_4)_2Ce(NO_3)_6$ | 3 | 0.18 | 0.08 |
| $FeCl_3.6H_2O$ | 3 | 0.16 | 0.007 |
| $FeCl_3$ (dry) | 4 | 0.15 | 0.008 |
| $Fe(ClO_4)_3$ | 4 | 0.16 | 0.01 |
| $H_2O_2$ | 5 | 0.16 | 0.03 |

Table 3 Electrical conductivity of polyaniline produced by $(NH_4)_2S_2O_8$ (HCl 0.1M) in different electric fields after 45 min

| Sample | Electric fields (KV/m) | Color change time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|--------|------|------|------|--------|
| 1 | 5 | 4 | 0.10 | 22 |
| 2 | 10 | 4 | 0.20 | 88 |
| 3 | 15 | 4 | 0.20 | 76 |
| 4 | 20 | 5 | 0.19 | 75 |
| 5 | 25 | 5 | 0.18 | 64 |
| 6 | 30 | 6 | 0.18 | 49 |

Table 4 Electrical conductivity of polyaniline produced by $(NH_4)_2S_2O_8$ (HCl 0.1M) in different limited electric fields after 45 min

| Sample | Electric fields (KV/m) | Color change time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|--------|------|------|------|--------|
| 1 | 8 | 3.5 | 0.18 | 73 |
| 2 | 9 | 4.0 | 0.20 | 78 |
| 3 | 10 | 4.0 | 0.20 | 88 |
| 4 | 11 | 4.5 | 0.19 | 86 |
| 5 | 12 | 4.5 | 0.18 | 80 |

Table 5 Electrical conductivity of polyaniline produced by $(NH_4)_2S_2O_8$ initiator and different solvents and in 10 KV/m electric field after 45 min

| Solvent/Dopant (0.1M) | Color change time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|---|---|---|---|
| $H_2O$/HCl | 4 | 0.20 | 89 |
| Ethanol/HCl | 4 | 0.07 | 22 |
| $CH_3CN$/HCl | 6 | 0.09 | 14 |
| THF/HCl | 5 | 0.15 | 14 |
| DMSO/HCl | 6 | 0.17 | 15 |
| m-Crosol/HCl | 4 | 0.20 | 11 |
| Acetone/HCl | 10 | 0.18 | 11 |
| $CH_2Cl_2$/PTSA | 45 | 0.11 | 0.001 |
| Benzene/PTSA | 45 | 0.11 | 0.003 |
| Nitrobenzene/PTSA | 15 | 0.05 | 0.042 |
| Cyclohexane/PTSA | 60 | 0.12 | 0.0004 |

Table 6 Electrical conductivity of polyaniline produced by $(NH_4)_2S_2O_8$ initiator and different dopants and in 10 KV/m electric field after 45 min

| Dopant (1M) | Color change time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|---|---|---|---|
| HCl | 4 | 0.20 | 120 |
| PTSA | 4 | 0.22 | 210 |
| DBSA | 5 | 0.24 | 500 |
| CSA | 5 | 0.23 | 320 |

Table 7 The electrical conductivities and molecular weight (from GPC method) in absence and presence (10 KV/m) of electric field for different conditions

| Condition of polymerization | Conductivity absence electric field (S/cm) | Molecular weight (GPC) absence electric field | Conductivity presence electric field (S/cm) | Molecular weight (GPC) presence electric field | $M_w/M_n$ |
|---|---|---|---|---|---|
| $H_2O$/HCl (0.1M) $(NH_4)_2S_2O_8$ | 10 | 530000 | 125 | 680000 | 2.3 |
| $H_2O$/HCl (0.1M) $(NH_4)_2Ce(NO_3)_6$ | 8 | 510000 | 110 | 650000 | 2.5 |
| $H_2O$/$Fe(ClO_4)_3$ | 0.01 | 450000 | 85 | 520000 | 2.1 |
| $H_2O$/DBSA (0.1M) $(NH_4)_2S_2O_8$ | 50 | 490000 | 510 | 655000 | 2.5 |

peroxydisulfate, $[(NH_4)_2S_2O_8]$, in different and limited electric fields after 45 min respectively. As shown in the Tables electrical conductivities will increase by increasing the electric field. But after about 10 KV/m, electrical conductivity has been decreased steadily. May be the reason is because of total dipole moment process decrease during a polymer chain. Considering this fact that each of two monomers has joined to each other with special angle, this reduction is natural. Results show at first electrical conductivities increased but approximately after 10 KV/m they decreased.

Table 5 shows electrical conductivity of polyaniline produced by $(NH_4)_2S_2O_8$ initiator in

different solvents and in 10 KV/m electric field after 45 min. Based on dielectric constant, solvent effect has been verified. It has been concluded that the best solvent would be water. It has been characterized due to its highest dielectric constant.

Hence, we improved electrical conductivities by different dopants which have been shown in Table 6. $M_w$ determinations by GPC were carried out on 0.05 wt./vol.% EB solutions in HPLC grade NMP. Visually, all the samples were totally soluble. EB powders were dissolved by stirring for 15 h, then the solutions were filtered. We determined the $M_W$ of each of synthesized polymers with high conductivities by GPC on EB solutions in NMP.

Table 8 Electrical conductivity of polyaniline produced by ($NH_4S_2O_8$) in different times and without magnetic field

| Sample | Time of polymerization (min) | Color change time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|--------|------------------------------|-------------------------|--------------------------------|--------------------------------|
| 1 | 10 | 3 | 0.007 | 0.0001 |
| 2 | 15 | 3 | 0.007 | 0.0004 |
| 3 | 20 | 3 | 0.009 | 0.008 |
| 4 | 30 | 3 | 0.010 | 0.009 |
| 5 | 40 | 3 | 0.010 | 0.009 |
| 6 | 60 | 3 | 0.011 | 0.008 |
| 7 | 120 | 3 | 0.012 | 0.008 |

Table 9 Electrical conductivity of polyaniline produced by ($NH_4S_2O_8$) in different magnetic field and after 45 min

| Sample | Magnetic fields (T) | Initiation of polymerization time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|--------|---------------------|------------------------------------------|--------------------------------|--------------------------------|
| 1 | 0 | 2 | 0.010 | 0.01 |
| 2 | 0.02 | 2 | 0.011 | 0.035 |
| 3 | 0.05 | 1.8 | 0.012 | 0.044 |
| 4 | 0.08 | 1.5 | 0.012 | 0.058 |
| 5 | 0.1 | 1.5 | 0.013 | 0.061 |
| 6 | 0.3 | 1.5 | 0.013 | 0.057 |
| 7 | 0.5 | 1.5 | 0.012 | 0.049 |

Table 10 Electrical conductivity of polyaniline produced by $FeCl_3.6H_2O$ in different times and without magnetic field

| sample | Time of polymerization (min) | Color change time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|--------|------------------------------|-------------------------|--------------------------------|--------------------------------|
| 1 | 10 | 6 | 0.001 | 0.00002 |
| 2 | 15 | 6 | 0.003 | 0.00008 |
| 3 | 20 | 6 | 0.006 | 0.0011 |
| 4 | 30 | 6 | 0.009 | 0.0014 |
| 5 | 40 | 6 | 0.009 | 0.0018 |
| 6 | 60 | 6 | 0.009 | 0.0021 |

Table 11 Electrical conductivity of polyaniline produced by $FeCl_3.6H_2O$ in different magnetic field and after 45 min

| sample | Magnetic fields (T) | Initiation of polymerization time (min) | Weight of produced polymer (g) | Electrical conductivity (S/cm) |
|--------|---------------------|-----------------------------------------|--------------------------------|--------------------------------|
| 1 | 0 | 6 | 0.009 | 0.002 |
| 2 | 0.02 | 6 | 0.013 | 0.0024 |
| 3 | 0.05 | 5 | 0.016 | 0.0032 |
| 4 | 0.08 | 4 | 0.019 | 0.0036 |
| 5 | 0.1 | 4 | 0.019 | 0.0038 |
| 6 | 0.3 | 4 | 0.019 | 0.0037 |
| 7 | 0.5 | 4 | 0.022 | 0.0036 |

Table 12 The electrical conductivities of polyaniline produced by $FeCl_3$ in absence and under magnetic field (MF) (0.1T), for different solvents after 45 min

| solvent | Electrical conductivity absence MF (S/cm) | Electrical conductivity under MF (S/cm) |
|---------|-------------------------------------------|-----------------------------------------|
| $H_2O$ | 0.0020 | 0.004 |
| Acetic acid | 0.0012 | 0.0004 |
| Ethanol | 0.0008 | 0.0018 |
| Acetonitrile | 0.0004 | 0.0004 |
| Acetone | 0.0004 | 0.0022 |
| Dichloroehtane | 0.0003 | 0.0004 |
| THF | 0.0003 | 0.0004 |

In Table 7, electrical conductivities and molecular weights (from GPC method) in the absence and presence (10 KV/m) of electric field are reported in different conditions of polymerization of aniline. We showed that the molecular weight of PANi which was prepared by oxidation of aniline in different conditions of polymerization was about $M_w = 450000$ and $680000$ without and under 10 KV/m electric field, respectively.

Increasing the electric field to 10 KV/m caused an increase of the $M_W$ of the synthesized polymers as seen by the increase of their masses. Polyaniline of higher molar mass and electrical properties are produced under electric fields.

The experimental results show that the magnetic field has an orientation effect on polyaniline chain too. There exists the interaction between free radical initiator, high dielectric solvent (water) and cation radicals of polyaniline chain due to the electrostatic interaction. The longer polymerization time and the higher magnetic field can lead to degradation of polyaniline films and decrease conductivity and molecular mass.

Table 8 - 12 show electrical conductivities of polyaniline produced by different conditions in absence and presence different magnetic fields. By comparison of produced results from the two variety field conditions, we observed high similarity in behaviour of results. The sufficient concentration of $FeCl_3$ can lead to the more distinct morphology of polyaniline films.

## 3.1 Electrorheological and magnetorheological mechanisms

The mechanisms of the electrorheological (ER) (Liu and Choi 2012) effect have been studied

intensively. Winslow (Winslow 1949) proposed that the ER effect was induced by the fibrillation of wet particles with a proper net charge in the insulating carrier liquid. The adsorbed water or surfactant in the particles was also considered necessary by many researchers (Kim *et al*. 1996), forming an adhesive water bridge between particles. An electric double layer model was also introduced based on wet ER particles (Klass *et al*. 1967). However, these mechanisms have become limited for anhydrous ER fluids.

The conduction model (Tang *et al*. 1995) is another highly recognized mechanism for an ER fluid, considering that the ER effect in a dc or a low frequency ac field was induced by mismatch of the conductivity of the particles and fluid medium. This model can predict the current density and field dependence of yield stress on the electric field for ER fluids with a highly concentrated particle volume fraction and high electric field strength. On the other hand, the model can be only used in situations where the particle microstructure was fully formed in an ER fluid.

Magnetorheological (MR) fluids (Park *et al*. 2010), composed of soft magnetic particles with little hysteresis suspended in nonmagnetic liquid medium, are phase controllable materials under an external magnetic field (Gomez-Ramirez *et al*. 2009). They can be rapidly and reversibly transformed from a fluid-like to a solid-like state within milliseconds by showing dramatic and tunable changes of several orders of magnitudes in rheological properties by controlling external magnetic field strength (Andrei *et al*. 2009). In the absence of an external magnetic field strength, the magnetizable particles are randomly dispersed in the medium and theMRfluid behaves similarly to a Newtonian fluid.

When a certain magnetic field strength is imposed, the dispersed particles build up the chain-like structure in the field direction due to an induced magnetic dipole–dipole interaction, along with their electrically analogous ER fluids under external electric fields (Fang *et al*. 2010). Compared to the ER fluids, typically possessing several kPa of yield stress (except special cases of giant ER fluids) (Zhang *et al*. 2009), MR fluids are known to demonstrate an apparent maximum yield stress up to 100 kPa depending on their composition, particle concentration and magnetic field strength (Phule *et al*. 1998), generally showing a Bingham plastic fluid behavior with a yield stress.

The MR fluids are differentiated with ferrofluids and ferromagnetic particle suspensions, in which the ferrofluids are suspensions of magnetic nanoparticles with sizes of about 10 nm in an appropriate carrier liquid like water, kerosene or various oils. Compared with magnetic particles in MR fluids, the magnetic field with each particle containing only a single magnetic domain in the ferrofluids is permanent and the nanoparticles rotate randomly under Brownian motion because of their small size (Gollwitzer *et al*. 2009). Therefore they keep their fluidity even if subjected to strong magnetic fields.

## 4. Conclusions

Application of a moderate external electric and magnetic fields increases the efficiency of polymerization and decreases the duration of polymerization process. Therefore, conductivity and molecular weight were increased, too.

Electrical conductivities of synthesized PANi of powder under the best fields are higher than 500 and 25 S/cm relative to 10 KV/m electric field and 0.1 T magnetic field, respectively. It shows an enhanced resistance to ageing too. The best times of polymerization are 20 - 45 and 45 min and the best initiators are ammonium peroxydisulfate and FeCl3.6H2O for electric and magnetic fields,

respectively. Therefore, the best solvent and dopant are water and DBSA for both. Thus the obtained polyaniline is probably less branched than standard polyaniline as indicated by: (i) a better solubility; (ii) an enhanced conductivities; (iii) an enhanced crystalline; (iv) an enhanced molecular weight (i.e., $M_w$), at the absence of electric field (using standard polyaniline).

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