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Conducting polymer gel: formation of a novel semi-IPN from polyaniline and crosslinked poly(2-acrylamido-2-methyl propanesulphonicacid)

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Abstract

In the present article, a simple two-step technique for synthesis of electrically conducting hydrogel is described. The synthesized hydrogel is originally a semi-interpenetrating polymer network (IPN) in which conducting polyaniline (linear) is entrapped within a crosslinked polyelectrolyte gel viz. poly(2-acrylamido-2-methyl propane sulphonic acid) (PAMPS). A conventional photo-polymerization technique was followed for synthesis of the PAMPS gel while aniline is in situ polymerized within the gel, giving rise to the desired semi-IPN. For comparison, a neutral gel based semi-IPN PAn–PAAm (polyaniline–polyacrylamide) was also prepared following the similar route. PAn imparts appreciable electrical conductivity to both PAMPS and PAAm based gels without hampering their existing properties that indicates the success of the work. Synthesis of the gel was studied in detail and different experimental conditions were optimized. Different physical properties of the gel viz. its degree of swelling, electrical conductivity, mechanical strength, etc were also studied. The resultant semi-IPN or the composite hydrogel was found to possess appreciable electrical conductivity, good swellability and mechanical strength. The electro-driven volume contraction of the composite gel was found to occur at sufficiently lower voltage compared to the pure PAMPS gel, which indicates its promising application aspect in fabrication of chemomechanical devices.

Keywords: Polyaniline; PAMPS; Polyelectrolyte gel

1. Introduction

Polymeric gels are defined as a system of crosslinked polymers (one or more) interstitial spaces of which are filled up with a fluid. The crosslink may be a weak physical link (provided by hydrogen bonding or electrostatic interaction) or a stable chemical bonding, depending upon which the gel is considered to be a physical gel or a chemical gel [1,2]. The system is obviously a soft and wet one having enough rigidity to hold a particular form and enough flexibility to undergo reversible deformation under different external stimuli (physical/chemical) [3]. Polymeric gels possess a very long history because there are lots of natural gels like polysaccharides, proteins, etc which are existing in living bodies and are used as food items. Nevertheless, polymeric gels till now are considered to be intelligent materials because of their interesting physical properties and wide-spread application potential. Commonly available polysaccharide gels viz, aggarose, carrageenan etc and synthetic gels prepared from PAMPS, polyacrylamide (PAAm), polyacrylic acid (PAA), copolymers of few liquid crystalline polymers etc, have shown highly important and interesting physical properties. Out of such properties, volume phase transition, high strength, shape memory properties, cooperative binding, low surface friction etc, have been successfully exploited in fabrication of gel based actuators, artificial muscles, sensors and different chemomechanical devices [4–9].

Inherently conducting polymers (ICP) present a group of conjugated organic polymers, namely polyaniline (PAn), polypyrrole (PPy), polythiophene (PTh), etc having high electronic conductivity [10,11]. Unusual electronic and optical properties of these polymers have drawn a good deal of interest over different fields of science and technology [12]. Over last two decades or more, there has been a

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continuous effort to explore different aspects of both conducting polymers and polymeric gel systems and suitable applications of these materials have been established. It is very surprising that these two materials (i.e. conducting polymers and polymeric gels) share few common fields of application like artificial muscles, sensors and actuators, controlled drug delivery, etc but effective combination of these two branches is not achieved yet. Polyelectrolyte gels (PAMPS, PAA) obviously possess ionic conductivity, which has been utilized in fabrication of chemomechanical devices with various kinds of motion [13–15]. Such electroactive polymeric (EAP) gels, however, are certainly devoid of electrical/electronic conductivity which have often caused serious difficulties like high operating voltage, poor response time, etc to the devices. Owing to the insolubility, intractability and few other difficulties, conducting polymers themselves cannot be turned to gels or cannot be combined with conventional polymers to form gels. However, success of such a combination is expected to improve different application aspects of the EAP gel systems especially those, which require electrical driving force. Improved response time and lower operating voltage is expected for a gel-based mechanochemical device if the gel possesses appreciable electrical conductivity [16].

PAn in its dedoped form is partially soluble in *N*-methyl pyrrolidone (NMP); the solution is spontaneously and irreversibly transferred to gel after some time [17,18]. However this kind of gelation is not controllable and therefore considered to be a hazard to the system. Besides there have been only few examples where polyaniline (PAn) and polythiophene (PTh) were turned to thermoreversible gels either by using some suitable solvent system or by the method of co-continuous blending [19–23]. For example, Ikkala et al. have selected dodecyl benzene sulphonic acid (DBSA) as suitable solvent as well as a dopant for formation of thermoreversible gel [19,20]. Almost same approach was followed by Nandi et al. [21,22] who have studied the effect of a series of sulphonic acids namely the camphor sulphonic acid (CSA), dinonylnaphthalene sulphonic acid (DNNSA), etc to form the PAn thermoreversible gel. In each case electrical conductivity was present to some extent in the gels. Synthesis of alkyl substituted PTh gel has been regularly cultivated by the research group of Ikkala, Inganas and Pepin-Donat et al. [23]. In all these examples, only the physical gel is obtained in which the improved solubility and thermal stability of conducting polymers as well as reversible formation of network is established; these materials, however, cannot possess the characteristic properties and application potential of conventional crosslinked gel systems. There are only few references in which conducting polymers are combined with crosslinked gels using some difficult chemical or electrochemical techniques [24,25]. Here we have synthesized electrically conductive hydrogels by chemical combination of PAn with a very familiar polyelectrolyte hydrogel viz. PAMPS. Prime objective of this work is to combine electrically conductive form of PAn with the conventional gel systems without hampering the characteristic physical properties of the latter. Synthesis technique and some preliminary investigations have indicated the success of the attempt, which will be presented here.

2. Experimental

2.1. Materials

Acrylamide and AMPS (monomer) were purchased from TCI, Japan. Aniline (monomer) and HCl were taken from Junsei Chemicals (Japan). Methylene-bis-acrylamide (MBAA the crosslinker), oxoglutaric acid (OGA, photoinitiator) and NaCl were supplied by Wako (Japan). Extra pure ammonium peroxodisulphate (APS, oxidizing agent for aniline polymerization) was purchased from Aldrich (USA). Double distilled water was used for different purposes and UV lamp was used for providing light of appropriate wavelength for photopolymerization.

2.2. Methods

2.2.1. Preparation of sample

Preparation of PAn-PAMPS composite gel is a two step process; at first the PAMPS gel is prepared following the UV-photo induced polymerization technique followed by polymerization of aniline within the first network. The first step is very similar to the free radical induced gel formation of PAMPS published earlier [26]. The modifications brought to the system were, to apply photo induced gel formation method and to add measured volume of aniline to the polymerizing solution. The aqueous solution of AMPS containing the crosslinker MBAA and photoinitiator OGA was taken within a cell made of two glass plates separated by a silicone spacer with required thickness. The cell was degassed and placed within a photo chamber full of nitrogen and subjected to exposure to UV radiation ($\lambda = 365$ nm). After 6 h the gel formation is complete and the gel is taken out from the cell for the second step of polymerization. At this state the gel contains aniline monomer dispersed within the PAMPS gel; the gel was cut into strips of different sizes and were immersed in the solution of APS in 1 N HCl (at room temperature) for aniline polymerization to take place. The gels are seen to swell slightly, and nucleation of polymerization is indicated by blue coloration within the transparent gel. Gradually the gel becomes green and after 45 min to 1 h, the green gels are taken out from the solution and immersed in 1 N HCl. A set of PAAm-PAn gels were also prepared following the identical technique, only the AMPS solution was substituted by AAm solution of same concentration.

2.2.2. Swelling measurements

Degree of swelling (*q*) of one set of PAMPS and PAMPS-PAn gel were calculated by the conventional method of weight gain using distilled water and NaCl solution of different concentrations [8].

2.2.3. Electrical conductivity

Electrical conductivity of these gels were measured by two probe method. Small pieces of gels cut from the strip were presses between two platinum foils and connected to a Hokuto Denko (HA-151) Potentiostat/Galvanostat for measurement.

2.2.4. Mechanical strength

Mechanical properties of the gel samples were measured by Tensilon Orientec RTC-1150A, an universal mechanical testing machine (interfaced with a computer) in two ways, by compression and by elongation. For compression, gels were cut into circular disks using a cutter and the strength was measured under compression till breaking against a stainless steel platform. For the elongation measurement, samples were cut from strips in dumbbell shape. The strips were fixed between two holders, drawn by a constant velocity, and the breaking strain and stress were recorded.

2.2.5. Gel contraction

The contractile behavior of the PAMPS-PAn gel was studied on a series of gel samples with surface area $10\times10\ \text{mm}^2$ and variable thickness. The piece of gel was placed within two Pt foils of same surface area attached with a micrometer (Peacock) in which the expansion or contraction is indicated by the movement of a pointer. The micrometer is attached to a function generator (HP 8116 A), a potentiostat (Hokuto Denko HA-151) and a recorder. Selected voltage (1–5 V) was applied and the corresponding thickness is recorded; the technique is similar to that followed earlier [27,28].

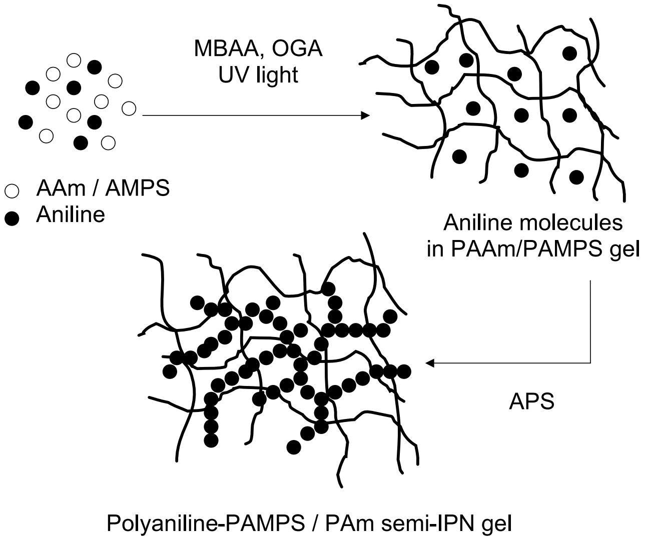
3. Results and discussions

3.1. Synthesis

It has already been mentioned that synthesis of an electrically conducting hydrogels with retention of other physical properties like reversible swelling, flexibility, etc is the prime objective of the present work. The gel we have prepared is a semi-IPN of PAMPS and PAn although the conventional simultaneous or sequential methods for synthesis of a semi-IPN or a double network (DN) gel could not be applied here. Primarily, we proceeded with the three-step process widely applied earlier for DN gel formation. This requires synthesis of the crosslinked PAMPS gel (step 1) and subsequent impregnation (step 2) of the gel in the solution of the second monomer (aniline). The gel is finally exposed to APS solution (step 3) in order to polymerize the aniline confined within the PAMPS gel. In this techniques two problems were encountered, firstly aniline could not properly penetrate into the depth of the gel and/or during the exposure of the gel to APS solution aniline leached out from the gel matrix and polymerized in solution. In order to avoid these problems we have modified the technique and started with a solution containing both aniline and AMPS. That is, instead of introducing aniline into the preformed gel, we have added this to the monomer (AMPS) solution prior to gel formation. The outline of the present synthesis technique is shown in the Scheme 1. In the first step AMPS is polymerized and subsequently crosslinked (by MBAA) to form the gel but aniline remains unchanged; aniline is polymerized at the second step i.e. on exposure to APS. It ensures homogeneous distribution of aniline within the gel matrix and lowers the chance of leaching out. Therefore, the present technique is a two-step process compared to the three-step conventional process. However, the gel can be termed as a semi-IPN only after completion of polymerization of aniline.

In order to investigate the effect of the polyelectrolyte nature of the gel, in a few points comparison with a neutral gel (PAAm) was done and a set of PAn-PAAm gels were also prepared following identical synthesis technique. It was observed that PAMPS itself being highly acidic matrix aniline molecules are much more firmly bound to PAMPS compared to that in PAAm; after exposure to APS, PAAm-aniline gel produces free polymer in larger amount than the PAMPS-aniline. If the surface of the gel is washed prior to the exposure to APS, the fraction of free PAn is decreased remarkably which reveals that the surface aniline molecules are prone to leaching out.

Thickness of the gel is also an important factor for the polymerization of aniline inside the gel. We have studied two types of gels, the thicker one (3 mm) and the thinner one (1 mm); for the thicker PAMPS gel polyaniline



Scheme 1. Two-step synthesis of PAn-PAMPS semi-IPN conducting gel.

concentration is very high on surface while at the center it is quite low. Un-uniform distribution of PAn in the gel could be easily visualized; on cutting the gel into pieces, the inner portion is found to be almost colourless in contrast to the dark green outer faces. However, for the thinner gel, presence of PAn is almost uniform throughout the gel indicated by the green colour of PAn in the interior. Using the 3 mm PAAm gel as matrix free PAn was produced in larger amount and the PAn distribution inside the gel was found to be quite inhomogeneous, changing the thickness of the gel to 1 mm homogeneity was improved. Concentration of both aniline and APS is also very important for successful synthesis of the conducting gel. In conventional technique, the optimum aniline; APS mole ratio for aniline polymerization was found to be 1:1.25, but in the present condition even the 1:1 ratio led to over-oxidation of polyaniline to black and insulating form and the optimum molar ratio was found out as 1:0.5. In the present system all aniline molecules are not equally exposed to the APS solution, only surface aniline molecules are initially in contact of APS. If the concentration of APS is very high with respect to the surface aniline molecules, over-oxidation invariably takes place. If the aniline concentration in the medium is very low, either it is not at all polymerized (if the APS is taken in proportionally smaller concentration polymerization proceeds only up to brown oligomeric stage) or it is over-oxidized very soon. On the other hand if the concentration of aniline is very high (and APS concentration is taken proportionally high) surface aniline molecules are polymerized rapidly and the pores of the gel are quickly blocked to prevent the diffusion of APS inside the gel. Therefore, it is obvious that in order to obtain the IPN gel in good condition (considering homogeneity, mechanical property and electrical conductivity) concentration of aniline and APS should be optimum and the surface should be washed lightly in order to prevent quick surface polymerization. The optimum composition, found out by trial and error was settled as 0.7 M aniline with 1 M AAm or AMPS solution (MBAA and OGA: 0.1 mol % of AMPS/AAM). During polymerization the solution was occasionally stirred in order to prevent local polymerization and to provide homogeneous exposure of the gel to APS. After completion of polymerization the green coloured gel was taken out, washed with 1 N HCl solution and immersed in the same solution to reach equilibrium condition.

It should be mentioned here that surface morphology of pure PAMPS and PAAM gels and those with PAn were studied using a Hitachi, Scanning Electron Microscope (225 ON) after coating with Pt. A typical SEM image of dried PAn-PAMPS semi-IPN hydrogel is shown in Fig. 1. However, no remarkable change in surface morphology was observed between pure PAMPS and the semi-IPN which can indicate that the polyaniline, in both systems was synthesized within the network of the gel; in other words, combination with PAn is not a surface modification. However, PAn affects bulk physical properties of the system that will be clarified from the further experiments and discussions.

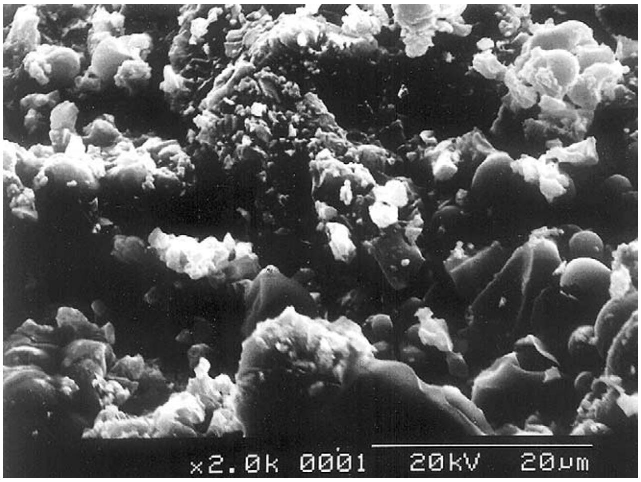


Fig. 1. Surface morphology of PAn-PAMPS semi-IPN hydrogel (1 mm) obtained by using SEM technique.

3.2. Degree of swelling (*q*)

Extent of swelling of PAMPS and PAn-PAMPS gels in water and different concentration of NaCl solution are listed in Table 1. The most remarkable observation is that absolute value of *q* is much higher in the PAn-PAMPS system as compared to the pure PAMPS. In the present system relatively large fraction of aniline (∼20% of the AMPS w/w) is present within the gel, which is readily converted to anilinium ion by the protons of AMPS. Anilinium ions are supposed to be gathered in the vicinity of the AMPS/PAMPS molecules and hamper the crosslinking of the PAMPS chains. Therefore, degree of crosslinking (DCL) is lowered in the composite gel system that is reflected in larger value of *q*.

It is also observed that for pure PAMPS gel degree of swelling (*q*) is abruptly lowered on changing the medium from distilled water to 0.001 M NaCl; the value of *q* decreases more than 50% in the latter. However, on further increasing the concentration of NaCl in the medium the value of *q* is only nominally lowered. On the other hand value of *q* in the PAn-PAMPS gel is monotonically lowered as the concentration of NaCl is gradually increased from 0.001 to 0.1 M.

3.3. Electrical conductivity

Electrical conductivity of PAMPS and PAn-PAMPS gels are shown in Table 1. Both the gels being very soft it is very difficult to obtain the absolute value of conductivity by the standard two probe method, only the approximate values could be obtained and reported here. In order to avoid water evaporation and consequent de-swelling the measurements were done in closed chamber. PAn-PAMPS gel possesses

Table 1
Results of different measurements done on PAMPS and PAAm based pure gels and PAn incorporated semi-IPNs

Sample	Concentration of NaCl solution (M)	Degree of swelling (%)	Electrical conductivity (S/cm)	Elongation at break (%)[a]	Breaking stress (MPa)[a]	Young's modulus (MPa)
PAMPS	0.000	98.00	$\leq 10^{-5}$	40.00 (3 mm)	0.012	0.030
	0.001	48.38				
	0.010	47.31		87.50 (1 mm)	0.045	0.051
	0.100	43.58				
PAn-PAMPS (High aniline loading)[b]	0.000	212.00	4.0	92.00 (3 mm)	0.014	0.0152
	0.001	134.63				
	0.010	83.12		37.50 (1 mm)	0.0376	0.100
	0.100	34.00				
PAn-PAMPS (surface washed)	–	–	0.2	41.90 (1 mm)	0.033	0.0787
PAAm[c]	–	–	–	278.29	0.0083	2.98×10^{-5}
PAn-PAAM	–	–	3.0	128.58	0.0067	5.21×10^{-5}
PAn-PAAM surface washed)	–	–	1.3×10^{-3}	121.49	0.0056	4.6×10^{-5}

[a] Measured by elongation technique.

[b] PAn-PAMPS (low aniline loading) shows degree of swelling 249 and 188.5 in pure water and NaCl solution (0.001 M).

[c] All measurements of PAAm and PAn-PAAm gel were done on samples with thickness 1 mm.

both ionic and electronic conductivity, ionic counterpart is contributed by PAMPS. It was observed that conductivity of pure PAMPS gel is only $\leq 10^{-5}$ S/cm, lower by an order of 10^4 as compared to the PAn-PAMPS composite ($\sim$0.21 S/cm). On the other hand pure PAAm is completely insulating (no ionic conductivity) although conductivity of PAn-PAAm composite is at least of the order of 10^{-3} S/cm. Therefore the contribution of ionic counterpart is almost negligible to the total conductivity of the gel; the observed conductivity in each of the samples is mainly electronic and accounted for the presence of PAn component.

Electrical conductivity of both thick (3 mm) and thin (1 mm) composite gels were measured. Thin samples of PAn-PAMPS possess higher conductivity because of the uniform dispersion of PAn throughout the sample compared to the thick gels inner portion of which is devoid of PAn. The samples with surface washed before exposure to APS (in order to avoid surface accumulation of PAn) were also subjected to measurements of electrical conductivity. The sample with surface not washed shows higher conductivity owing to the accumulation of free PAn on surface. For PAn-PAAm systems absolute value of conductivity is much lower compared to PAMPS based samples, conductivity of the surface washed samples are significantly lower than others. It should be mentioned here that for the highly acidic and anionic nature of the supporting gel anilinium cations are firmly attached to the PAMPS matrix compared to that in PAAm. This is the reason for higher PAn loading in samples resulting in higher electrical conductivity.

3.4. Mechanical property measurements

It should be mentioned at first that mechanical strength measurements under compression requires a minimum thickness around 2 mm. Therefore, thin samples (1 mm) made of both PAMPS and PAAm could not be subjected to compression. Results of mechanical strength measurements of PAMPS and PAn-PAMPS gel samples (3 mm) under both compression and elongation are shown in Fig. 2(A) and (B) and the respective results are presented in Table 1. PAn itself is a polymer with highly stiff backbone, however, this polymer is found to have different effect on the mechanical properties of the composite gels with different thickness. From the respective elongation at break and breaking modulus values of PAMPS and PAn-PAMPS gels (3 mm) it is observed that in this case stiffness of PAn-PAMPS gel is lowered after introduction of PAn which is graphically shown in Fig. 2(A). This conclusion is further supported by the results of the compression measurement as shown in Fig. 2(B). However, this effect is completely reversed for the thin gels samples as shown in Fig. 3(A), here introduction of PAn results in remarkable increase of stiffness of the samples as is obvious from modulii values at breaking point. It has been already discussed that for the former set of samples (3 mm) PAn is deposited mainly near the surface of the gel leaving the bulk almost unchanged, therefore stiffness of PAn component is not imparted to the gel. Rather presence of sufficient amount of aniline monomer (20% w/w of the AMPS) during synthesis of the gel hampers the crosslinking of PAMPS to some extent for which the elongation at break increases and breaking modulus is lowered. This interpretation is supported from the results of the swelling measurements of gels discussed earlier, swellability of the thicker composite gels is much higher compared to the pure PAMPS gel which also indicates lower DCL of the former. In the thin PAMPS gel the stiffness is somewhat increased compared to the thicker one which may be due to the difference in DCL.

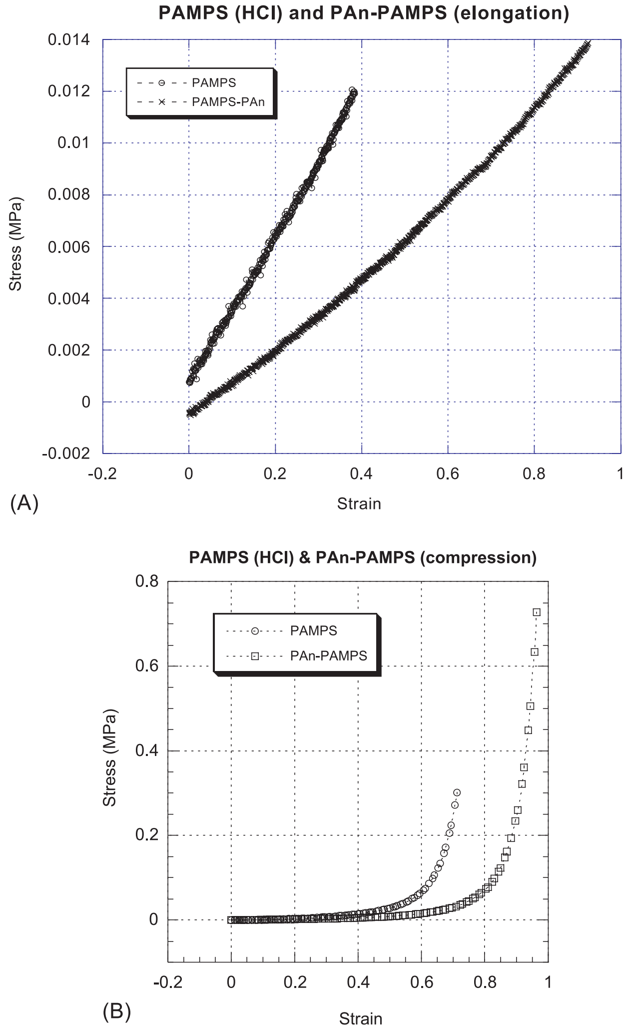


Fig. 2. (A) Stress–strain behavior of PAMPS (in HCl) and PAn-PAMPS (in HCl) gel samples (thickness 3 mm) measured by elongation method. (B) Stress-strain curve obtained for PAMPS (in HCl) and PAn-PAMPS (in HCl) gel samples (thickness 3 mm) measured by compression method.

However, in the thin semi-IPN samples PAn is almost homogeneously distributed within the matrix so that after introduction of PAn stiffness of the samples increases. If the surface of the gel (containing aniline monomer) is washed prior to polymerization of aniline, surface deposition of PAn is decreased to some extent with parallel lowering of the stiffness of the gel.

As the experiment is repeated with thin PAAm gels it was observed that elongation of PAAm gel is much larger compared to both PAMPS and PAn-PAMPS gels. Stiffness of PAAm gel is slightly increased after inclusion of PAn although the mechanical profile remains almost unchanged as shown in Fig. 3(B). Stiffness of the surface washed sample is again found to be lower to a small extent. This result is in pace with the earlier results and the corresponding explanation that in highly acidic PAMPS matrix aniline molecules and PAn chains are more firmly anchored than in PAAm resulting in extensive modification of the matrix. Therefore, it can be interpreted that both PAMPS (polyelectrolyte) and PAAm (neutral) gels can provide good support matrix for PAn and in both instances stiffness of the gel increases after PAn inclusion. However, depending upon the nature of interaction of PAn with the matrix extent of increase is different in two cases.

3.5. Electrocontractile behavior

It has been elaborately explored earlier that polyelectrolyte gels undergo expulsion of water and corresponding shrinkage near anode under applied potential [27,28]. This is an outcome of electrophoretic effect by which the hydrated protons (cations) move towards cathode along with the attached water molecules and water migration from anode (as derived from Flory's theory [29]) takes place. In the present experiment we have fixed the gel within two Pt electrodes and applied dc voltage across the electrodes. It was observed that up to 3 V/cm there is no change of thickness of the gel but at 4 V/cm thickness of the gel starts to be lowered. The conducting gel can undergo profuse water expulsion and volume contraction at ∼5 V/cm, which is in sharp contrast to the electrically insulating gels that needs potential as high as ∼10–30 V/cm or so for electrocontraction to take place [30–32]. This result indicates importance of the conducting gel in fabricating electromechanical devices with lower energy consumption.

4. Conclusion

In the present article synthesis and characterizations of a conducting polymer based polyelectrolyte gel is reported that possesses both ionic and electronic conductivity. It can be concluded that PAMPS can provide an excellent matrix for PAn so that it can introduce electronic conductivity to the system without hampering its existing properties. However, the gel matrix should be sufficiently thin (∼1 mm) in order to attain homogeneous distribution of PAn throughout the gel. Characterizations have confirmed that PAn is present in the system in chemically pure protonated (emeraldine) form with retention of its electronic conductivity. Due to the presence of PAn, stiffness of the gel is somewhat enhanced although its electrocontractile behavior is not hampered. At the same time the phenomenon of contraction is found to take place at much lower potential that is helpful for easier operation of the gel-based devices. Taking the gel in rectangular slab asymmetric contraction is observed; if the gel is taken in spherical form the symmetric contraction is expected that will be revealed in our further investigation. In the present article optimization of synthesis conditions and characterizations have been emphasized and only few preliminary results on the electrocontractile behavior has been included. Elaborate studies on electrocontraction are under progress.

Combination of PAMPS with PAn is not certainly a

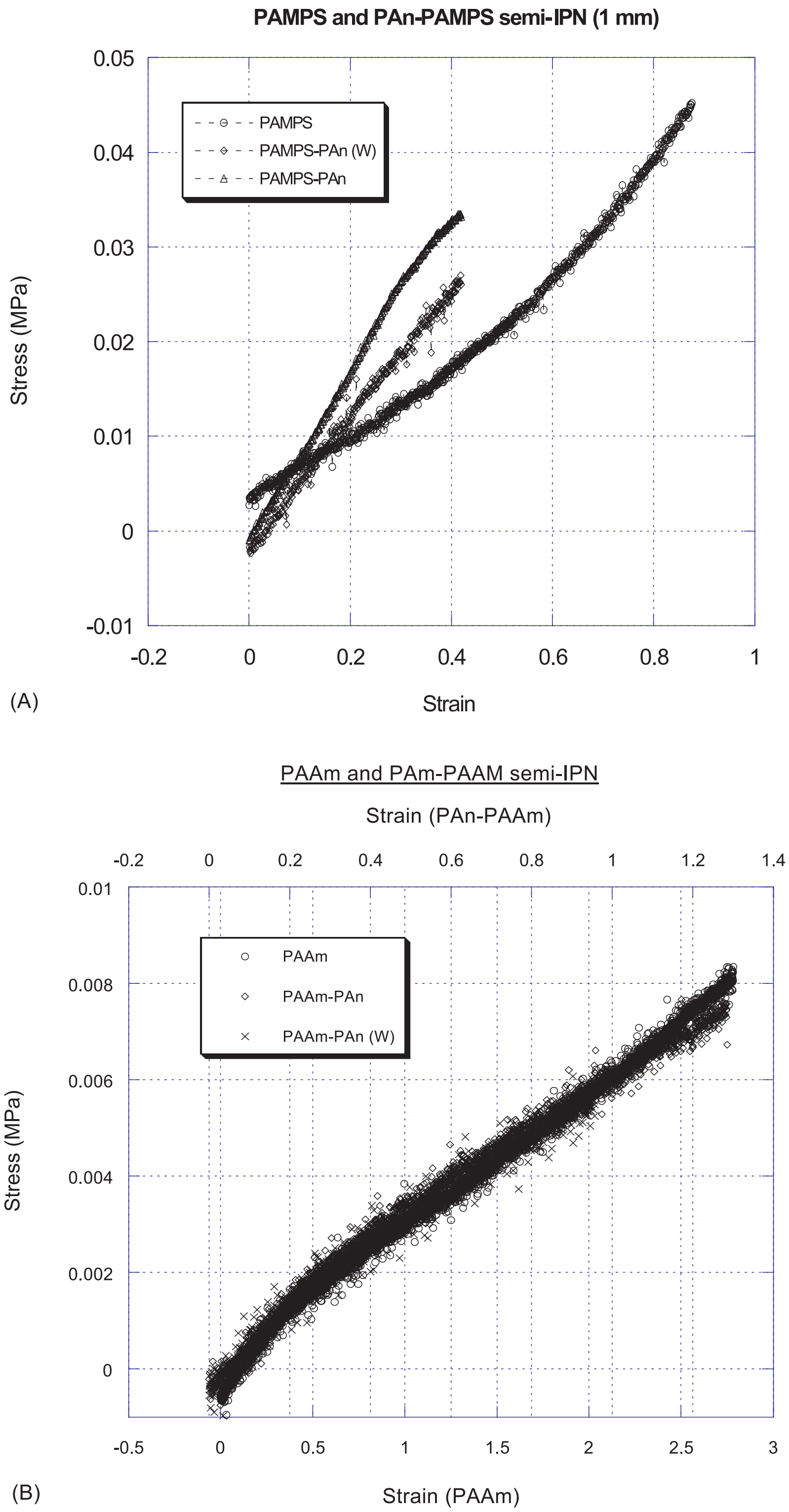


Fig. 3. (A) Elongational behavior of thin samples (1 mm) of PAMPS, PAn-PAMPS semi-IPN and PAn-PAMPS semi-IPN with washed surface (W). (B) Elongational behavior of thin samples (1 mm) of PAm, PAn-PAAm semi-IPN and PAn-PAAm semi-IPN with washed surface (W).

surface modification because surface morphology of the composite is not much affected by this modification. However, larger fraction of PAn is accumulated near the surface if the gel is not thin enough. Following electrosynthesis of PAn within the gel matrix a more accurate control over the system can be attained which raises the interest for further investigation. Earlier we have studied electrochemical synthesis of PPy within crosslinked matrix of PVA so as to synthesize a semi-IPN with novel properties [33]. Similar technique can be followed for synthesis of ICP based IPN or semi-IPN hydrogels in future. The neutral matrix (PAAm) has also been proved to be highly suitable for supporting PAn chains but this system was not elaborately studied in this context. It will also be explored in our forthcoming publications.

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