

# Diazotization of Anilines and Diazo Coupling with a Coupling Component Mediated by a Polymer-Supported Sodium Nitrite and a Polymeric Acid

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**ABSTRACT:** Polymer-supported reagents have become the subject of considerable and increasing interest as insoluble materials in the organic synthesis. In this study, diazonium salts are generated and are coupled with a coupling component by using a polymer supported nitrite and a polymeric acid. In this procedure, the azo chromo-

phores are formed in a clean and efficient manner, the work-up is easy and yields are high to excellent. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 123: 2762–2767, 2012

**Key words:** diazotisation; azo chromophore; amine; polymer-supported reagent

## INTRODUCTION

Diazonium salts are useful synthetic building blocks in organic synthesis as these compounds can be linked to methine or aromatic sp<sup>2</sup>-hybridized C-atom to obtain azo chromophores. Azo compounds are very important in the field of dyes,<sup>1</sup> and are widely used as dyes and analytical reagents. They can also be used as indicators in chemical laboratories and as biological stains. On the other hand, azo compounds have more recently found potential applications in various fields such as biomedicine<sup>2,3</sup> and organic synthesis.<sup>4,5</sup> They also have excellent optical and photoelectric properties.<sup>6–8</sup>

There are many methods available for the synthesis of azo compounds.<sup>9–13</sup> Synthesis of azo derivatives can be accomplished by the reduction of nitro aromatics with metal hydrides,<sup>14,15</sup> zinc in strongly alkaline media,<sup>16</sup> lead and triethylammonium formate,<sup>17</sup> active-iron base reducing system,<sup>18</sup> or dicobalt octacarbonyl.<sup>19</sup> Many other methods for preparation of azo compounds have been described in the literature.<sup>4,5,13,20</sup> Some of them suffer from low yields and undesired side reactions. In addition, they may also require harsh conditions or can generate dangerous pollutants for the environment.<sup>20</sup> Consequently, new methodologies with

milder reaction conditions, and simply work-up are welcomed.

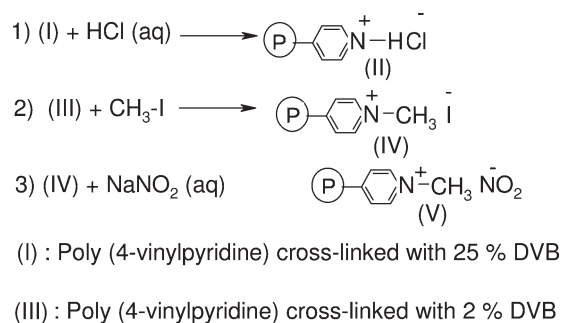
Although polymer-supported reagents especially anion exchange resins have been widely applied in organic synthesis,<sup>21–39</sup> there are only a few reports in the literature for the synthesis of azo compounds based on polymeric acid or polymer-supported reagents under heterogeneous conditions.<sup>40–45</sup>

Recently, we used crosslinked poly(*N*-methyl-4-vinylpyridinium) nitrite, [P<sub>4</sub>-VP]NO<sub>2</sub>, for synthesis of nitroalkanes,<sup>29</sup> *N*-nitrosation of secondary amines,<sup>30</sup> as a heterogeneous system and solvent free *N*-nitrosation of secondary amines by grinding.<sup>31</sup>

In continuation of our efforts to develop application of [P<sub>4</sub>-VP]NO<sub>2</sub> in organic synthesis, the study of diazotisation and diazocoupling reactions of aniline and its substituted derivatives with a coupling agent such as phenol, 2-naphthol, anisole, and *N,N*-dimethylaniline over [P<sub>4</sub>-VP]NO<sub>2</sub> and [P<sub>4</sub>-VP]HCl under completely heterogeneous conditions are investigated. It is expected that the [P<sub>4</sub>-VP]HCl would be a proton source similar to all of the reported acidic solid supports or acidic resins such as polystyrene sulfonic acid and Nafion-H.<sup>45</sup> Therefore, the use of this acidic resin as a new solid acid for the generation of HNO<sub>2</sub> when used in conjunction with [P<sub>4</sub>-VP]NO<sub>2</sub> was tested.

Herein, we wish to report an efficient and a general method for diazotization and diazocoupling reactions of aniline and its substituted derivatives with a coupling component such as phenol, 2-naphthol, anisole, and *N,N*-dimethylaniline over [P<sub>4</sub>-VP]

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Scheme 1

NO<sub>2</sub>/[P<sub>4</sub>-VP]HCl under completely heterogeneous conditions.

## EXPERIMENTAL

### General

Chemicals were purchased from Fluka, Merck, and Aldrich chemical companies. Poly(4-vinylpyridine) crosslinked with 2% divinylbenzene (DVB), [P<sub>4</sub>-VP] 2% DVB, and poly(4-vinylpyridine) crosslinked with 25% DVB, [P<sub>4</sub>-VP] 25% DVB was purchased from Fluka chemical company (Germany).

Crosslinked poly(4-vinylpyridine) supported hydrochloric acid [P<sub>4</sub>-VP]HCl from [P<sub>4</sub>-VP] 25% DVB and crosslinked poly(4-vinylpyridine) supported sodium nitrite, [P<sub>4</sub>-VP]NO<sub>2</sub> from [P<sub>4</sub>-VP] 2% DVB were prepared according to our procedure (Scheme 1).<sup>29–31</sup> Progress of the reaction was followed by thin layer chromatography (TLC) using silica gel Poly Gram SIL G/UV 254 plates. Products were characterized by comparison of their Fourier transform infrared (FTIR), proton, and 13 carbon nuclear magnetic resonance (<sup>1</sup>H- and <sup>13</sup>C-NMR) spectra and physical data with pure compounds.

FTIR spectra were obtained by using a Bruker, Equinox (model 55), and NMR spectra were recorded on a Bruker AC 500, Avance DPX spectrophotometer at 500 MHz for <sup>1</sup>H and at 125 MHz for <sup>13</sup>C NMR in CDCl<sub>3</sub> solutions (using tetramethylsilane as an internal reference).

### Preparation of [P<sub>4</sub>-VP]HCl

To a concentrated hydrochloric acid, (10 mL) [P<sub>4</sub>-VP] 25% DVB (1 g) was added, and stirred for 24 h. The mixture was filtered, washed with distilled water until the filtrate gave a negative test for HCl. Then it was dried in vacuum at 50°C for 5 h to give 1.2 g of [P<sub>4</sub>-VP]HCl (II). The capacity of the polymer was determined by potentiometric titration with a 0.1 N solution of sodium hydroxide. The obtained activity of polymer was 5 mmol/g of the polymer.

### Preparation of [P<sub>4</sub>-VP]NO<sub>2</sub>

[P<sub>4</sub>-VP] 2% DVB (2.0 g) was treated with methyl iodide (5 mL) in acetonitrile (10 mL) and stirred for 24 h, at room temperature. The mixture was filtered and washed with distilled water and acetonitrile. It was then dried under vacuum in the presence of P<sub>2</sub>O<sub>5</sub> at 40°C to obtain crosslinked poly(*N*-methyl-4-vinylpyridinium) iodide, [P<sub>4</sub>-VP]I, (3.75 g).

To 20 mL of a three molar solution of sodium nitrite, 2.0 g of [P<sub>4</sub>-VP]I was added and slowly stirred for 24 h. The mixture was filtered off and washed rapidly with distilled water until the filtrate gave a negative test for NO<sub>2</sub><sup>-</sup>. It was then washed with ether and dried under vacuum in the presence of P<sub>2</sub>O<sub>5</sub> at 40°C, to obtain 1.1 g of [P<sub>4</sub>-VP]NO<sub>2</sub>.

The activity of this polymer was determined by potentiometric titration with a 0.1 N solution of silver nitrate. The obtained activity of polymer was 2.2 mmol/g of the polymer.

### General procedure for synthesis of azo dyes

To a mixture of primary aromatic amine (1 mmol), [P<sub>4</sub>-VP]HCl (0.4 g, 2 mmol), and acetonitrile (5 mL) in a round-bottomed flask (50 mL) 0.9 g of [P<sub>4</sub>-VP]NO<sub>2</sub> (1 mmol) was added, and while keeping the suspension at 0–5°C, it was stirred for 30 min.

A solution of 1 mmol of a coupling component in 5 mL of acetonitrile was added slowly with a dropper to the arenediazonium salt solution, whereas the mixture was well-stirred during addition. When the addition was completed stirring was continued for the time specified in Table I until a yellow–orange–red color appeared. Reaction monitoring was accomplished by TLC with ethyl acetate/*n*-hexane (30/70) as eluent. After completion of the reaction, the suspension was filtered and the solvent was evaporated. In general, the crystalline yellow–orange–red solid products were obtained in high yields (Table I).

If further purification was needed, flash chromatography on silica gel [eluent: ethyl acetate/*n*-hexane (30/70)] was used which provides highly pure products.

### Synthesis of 1-(phenylazo)-2-naphthol

To a mixture of aniline (93 mg, 1 mmol), [P<sub>4</sub>-VP]HCl (0.4 g, 2 mmol), and acetonitrile (5 mL) in a round-bottomed flask (50 mL), 0.45 g of [P<sub>4</sub>-VP]NO<sub>2</sub> (1 mmol) was added, and while keeping the suspension at 0–5°C, was stirred for 30 min. The slightly turbid pale grey solution as a benzenediazonium salt solution was obtained. Then the solution of 144 mg of 2-naphthol in 5-mL acetonitrile was added slowly with a dropper to the

**TABLE I**  
**Synthesis of Azo Chromophores Mediated by [P<sub>4</sub>-VP] HCl/[P<sub>4</sub>-VP] NO<sub>2</sub><sup>a</sup> in Acetonitrile at 0–5°C**





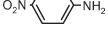
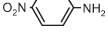



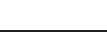
Entry	Ar-NH <sub>2</sub>	Ar-H	Product <sup>b</sup>	Time (h)	Yield <sup>c</sup> (%)
1	Ph-NH <sub>2</sub>	Ph-OH		12	94
2	Ph-NH <sub>2</sub>	2-Naphthol		12	95
3	Ar-NH <sub>2</sub>	Anisole	No reaction	15	0.0
4	Ar-NH <sub>2</sub>	<i>p</i> -nitro phenol	No reaction	15	0.0
5	Ph-NH <sub>2</sub>	Ph-N(Me) <sub>2</sub>		12	92
6	Ph-NH <sub>2</sub>	Hydroquinone		15	99
7		Ph-OH		13	87
8		2-Naphthol		14	89
9		Anisole	No reaction	15	0.0
10		<i>p</i> -nitro phenol	No reaction	15	0.0
11		Ph-N(Me) <sub>2</sub>		16	72
12		Ph-OH		13	82
13		2-Naphthol		14	90
14		Anisole	No reaction	15	0.0
15		<i>p</i> -nitro phenol	No reaction	15	0.0
16		Ph-N(Me) <sub>2</sub>		16	73
17		Ph-OH		10	98
18		2-Naphthol		10	97
19		Anisole	No reaction	15	0.0
20		<i>p</i> -nitro phenol	No reaction	15	0.0
21		Ph-N(Me) <sub>2</sub>		10	98

<sup>a</sup> Molar ratio of [P<sub>4</sub>-VP] HCl: [P<sub>4</sub>-Me] NO<sub>2</sub>: amine equal to 2 : 1 : 1.

<sup>b</sup> The structures were confirmed by comparison of the melting point, FTIR and <sup>1</sup>H-NMR spectra with those of authentic specimen.

<sup>c</sup> Isolated yields.

**TABLE II**  
Diazotization and Diazo Coupling Reactions in Different Reported Methods

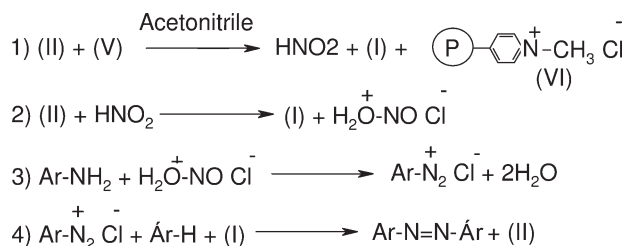
Entry	Ar-NH <sub>2</sub>	Ár-H	Reaction condition	Yield <sup>a</sup> (%)	Ref.
1	Ph-NH <sub>2</sub>	Ph-OH	$\text{Ar-NH} + \text{Ár-H} \xrightarrow[\text{Clay (K10), 0-5}^\circ\text{C}]{\text{aq NaNO}_2, 2\text{h}} \text{Ar-N=N-Á}$	91	48
2	Ph-NH <sub>2</sub>	Ph-OH	$\text{Ar-NH} + \text{Ár-H} \xrightarrow[\text{HCl (aq), 0-5}^\circ\text{C}]{\text{Amberlyst-NO}_2^-} \text{Ar-N=N-Á}$	90 <sup>b</sup>	40
3	Ph-NH <sub>2</sub>	Ph-OH	$\text{Ar-NH}_2 \xrightarrow[\text{r.t., grinding}]{\text{NaNO}_2, \text{wet SSA}^b} \text{ArN}_2^+ \text{-OSO}_3\text{-SiO}_2 \xrightarrow[\text{r.t., grinding}]{\text{Ár-H}} \text{Ar-N=N-Á}$	Trace	41
4	Ph-NH <sub>2</sub>	Ph-OH	$\text{Ar-NH}_2 + \text{Ár-H} \xrightarrow[\text{Acetonitrile, 0-5}^\circ\text{C}]{[\text{P}_4\text{-VP}]\text{HCl} / [\text{P}_4\text{-Me}]\text{NO}_2} \text{Ar-N=N-Ár}$	94	Entry 1, in Table 1
5	Ph-NH <sub>2</sub>	Ph- NMe <sub>2</sub>	$\text{Ar-NH} + \text{Ár-H} \xrightarrow[\text{Clay (K10), 0-5}^\circ\text{C}]{\text{aq NaNO}_2, 2\text{h}} \text{Ar-N=N-Á}$	92	48
6	Ph-NH <sub>2</sub>	Ph- NMe <sub>2</sub>	$\text{Ar-NH} + \text{Ár-H} \xrightarrow[\text{HCl (aq), 0-5}^\circ\text{C}]{\text{Amberlyst-NO}_2^-} \text{Ar-N=N-Á}$	92 <sup>c</sup>	40
7	Ph-NH <sub>2</sub>	Ph- NMe <sub>2</sub>	$\text{Ar-NH}_2 \xrightarrow[\text{r.t., grinding}]{\text{NaNO}_2, \text{wet SSA}^b} \text{ArN}_2^+ \text{-OSO}_3\text{-SiO}_2 \xrightarrow[\text{r.t., grinding}]{\text{Ár-H}} \text{Ar-N=N-Á}$	81	41
8	Ph-NH <sub>2</sub>	Ph- NMe <sub>2</sub>	$\text{Ar-NH}_2 + \text{Ár-H} \xrightarrow[\text{Acetonitrile, 0-5}^\circ\text{C}]{[\text{P}_4\text{-VP}]\text{HCl} / [\text{P}_4\text{-Me}]\text{NO}_2} \text{Ar-N=N-Ár}$	92	Entry 5, in Table 1
9		Ph-OH	$\text{Ar-NH} + \text{Ár-H} \xrightarrow[\text{Clay (K10), 0-5}^\circ\text{C}]{\text{aq NaNO}_2, 2\text{h}} \text{Ar-N=N-Á}$	81	48
10		Ph- NMe <sub>2</sub>	$\text{Ar-NH} + \text{Ár-H} \xrightarrow[\text{HCl (aq), 0-5}^\circ\text{C}]{\text{Amberlyst-NO}_2^-} \text{Ar-N=N-Á}$	quant <sup>c</sup>	40
11		Ph- NMe <sub>2</sub>	$\text{Ar-NH}_2 \xrightarrow[\text{r.t., grinding}]{\text{NaNO}_2, \text{wet SSA}^b} \text{ArN}_2^+ \text{-OSO}_3\text{-SiO}_2 \xrightarrow[\text{r.t., grinding}]{\text{Ár-H}} \text{Ar-N=N-Á}$	83	41
12		Ph- NMe <sub>2</sub>	$\text{Ar-NH}_2 + \text{Ár-H} \xrightarrow[\text{Acetonitrile, 0-5}^\circ\text{C}]{[\text{P}_4\text{-VP}]\text{HCl} / [\text{P}_4\text{-Me}]\text{NO}_2} \text{Ar-N=N-Ár}$	98	Entry 21, in Table 1
13		Ph-OH	$\text{Ar-NH} + \text{Ár-H} \xrightarrow[\text{HCl (aq), 0-5}^\circ\text{C}]{\text{Amberlyst-NO}_2^-} \text{Ar-N=N-Á}$	80 <sup>b</sup>	40
14		Ph-OH	$\text{Ar-NH}_2 \xrightarrow[\text{r.t., grinding}]{\text{NaNO}_2, \text{wet SSA}^b} \text{ArN}_2^+ \text{-OSO}_3\text{-SiO}_2 \xrightarrow[\text{r.t., grinding}]{\text{Ár-H}} \text{Ar-N=N-Á}$	60	41
15		Ph-OH	$\text{Ar-NH}_2 + \text{Ár-H} \xrightarrow[\text{Acetonitrile, 0-5}^\circ\text{C}]{[\text{P}_4\text{-VP}]\text{HCl} / [\text{P}_4\text{-Me}]\text{NO}_2} \text{Ar-N=N-Ár}$	98	Entry 17, in Table 1
16		Ph- NMe <sub>2</sub>	$\text{Ar-NH} + \text{Ár-H} \xrightarrow[\text{HCl (aq), 0-5}^\circ\text{C}]{\text{Amberlyst-NO}_2^-} \text{Ar-N=N-Á}$	0.0	40
17		Ph- NMe <sub>2</sub>	$\text{Ar-NH}_2 + \text{Ár-H} \xrightarrow[\text{Acetonitrile, 0-5}^\circ\text{C}]{[\text{P}_4\text{-VP}]\text{HCl} / [\text{P}_4\text{-Me}]\text{NO}_2} \text{Ar-N=N-Ár}$	72	Entry 11, in Table 1
18		Ph-OH	$\text{Ar-NH} + \text{Ár-H} \xrightarrow[\text{HCl (aq), 0-5}^\circ\text{C}]{\text{Amberlyst-NO}_2^-} \text{Ar-N=N-Á}$	62 <sup>b</sup>	40

<sup>a</sup> The yields refer to the isolated pure products.

<sup>b</sup> Yields are based on the amount of phenolic derivative used.

benzenediazonium salt solution, whereas the mixture was well stirred during addition. When the addition was completed, stirring was continued for

12 h until an orange-red color appeared. Reaction monitoring was accomplished by TLC with ethyl acetate/n-hexane (30/70) as eluent. After completion



Scheme 2

of the reaction, the suspension was filtered and the solvent was evaporated. The crystalline orange-red solid product was obtained in 95% yield. M.P: 130–132 (lit <sup>46</sup>: 131–133), FTIR (KBr)  $\nu$  (cm<sup>-1</sup>): 3434 (NH/OH, intermolecular hydrogen bonding), 3031 (C–H), 1617, 1554, 1498, and 1447 (C=C), 1207 (C–O), 1261 (N=N), 751 and 839 (=C–H, bending out of plane). <sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$ : 16.050 (NH, s), 6.68 (1H, d, *J* = 9.2 HZ) 7.56 (1H, d, *J* = 9.2 HZ), 7.42 (1H, m), 7.21 (1H, t), 7.36 (1H, t), 8.37 (1H, d, *J* = 8 HZ), 7.58 (1H, d, *J* = 8.4 HZ), 7.3 (1H, t), and 7.11 (1H, t).

## RESULTS AND DISCUSSION

Crosslinked poly(4-vinylpyridine) supported hydrochloric acid, [P<sub>4</sub>-VP]HCl, and crosslinked poly(4-vinylpyridine) supported sodium nitrite, [P<sub>4</sub>-VP]NO<sub>2</sub>, were easily prepared according to our procedure (Scheme 1).<sup>29–31,39</sup> The hydrochloride salt of a crosslinked macroreticular poly(4-vinylpyridine) resin crosslinked with 25% DVB, [P<sub>4</sub>-VP]HCl, in bead form is an effective, yet mild acid for diazotization of aniline in the presence of polymer supported sodium nitrite, [P<sub>4</sub>-VP]NO<sub>2</sub> and diazocoupling reactions of aniline and its substituted derivatives with a coupling component (Scheme 2). Primary aromatic amine in the presence of [P<sub>4</sub>-VP] HCl/[P<sub>4</sub>-VP]NO<sub>2</sub> is converted to corresponding diazonium salts.

Aniline-based diazo component was prepared by coupling of diazonium salt with a coupling component. The reaction is believed to follow the typical pathway shown in Scheme 2.

To find out the best solvent for the diazotization and diazocoupling reactions of aromatic amines with a coupling component over [P<sub>4</sub>-VP]NO<sub>2</sub>/[P<sub>4</sub>-VP]HCl, aniline as a amine and phenol as a coupling component are chosen as model substrate. For optimization, it was treated with 1 mmol of aniline, 1 mmol of phenol, 1 mmol of [P<sub>4</sub>-VP]NO<sub>2</sub> and 2 mmol of [P<sub>4</sub>-VP]HCl in various available solvents at 0–5°C. Acetonitrile was proved to be the solvent of choice among other organic solvents.

Several primary amines such as aniline and substituted anilines were used for synthesis of the corre-

sponding diazonium salts by using [P<sub>4</sub>-VP]HCl/[P<sub>4</sub>-VP]NO<sub>2</sub> in acetonitrile. Also several different coupling component such as phenol, *p*-nitrophenol 2-naphthol, anisole, hydroquinone, and *N,N*-dimethylaniline were used for coupling with diazonium salts to obtain corresponding azo chromophores. The results and reaction conditions are summarized in Table I. The chemoselectivity of the method is also noteworthy. Although the diazonium salt derived from anilines was coupled with some coupling components such as phenol, 2-naphthol and *N,N*-dimethylaniline in high to excellent yields (72–99%), but an attempt to couple it with anisole and *p*-nitrophenol gave no products (Table I).

As seen in Table I, electron withdrawing group such as –NO<sub>2</sub>, in *p*-position of aniline increase its reactivity toward coupling with a coupling component (entries 17, 18, and 21 in Table I), whereas the presence of electron releasing groups such as –OCH<sub>3</sub>, and –OPh decreases their reactivity toward diazocoupling reactions (entries 7, 8, 11–13, and 16 in Table I). These results are in accordance with data for the relative rates of coupling for diazonium salts of aniline derivatives: 4-NO<sub>2</sub> : H : 4-CH<sub>3</sub>O 1300 : 1 : 0.1.<sup>47</sup> Also, these results are in accordance with the previously reported results by Ley and coworkers<sup>40</sup> To highlight the notable features of the presented protocol for synthesis of azo chromophores by using [P<sub>4</sub>-VP]HCl/[P<sub>4</sub>-VP]NO<sub>2</sub>, with some of those reported in the literature is compared (Table II). In Table II, the reaction conditions of some different reported methods for synthesis of azo chromophores are summarized.

## CONCLUSIONS

Poly(4-vinylpyridine) supported sodium nitrite and poly(4-vinylpyridine) supported hydrochloric acid were easily prepared, and used for preparation of different diazonium salts from primary aromatic amines in acetonitrile. After coupling, the diazonium salt with coupling component, azo chromophores were synthesized. With the ever increasing environmental restrictions and control on the release of effluent, the capability of this process which provides an efficient and a general synthesis of the azo compound is demonstrated.

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