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Controllable stereoselective synthesis of *cis* or *trans* pyrano and furano tetrahydroquinolines: Polyaniline-*p*-toluenesulfonate salt catalyzed one-pot aza-Diels–Alder reactions

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ABSTRACT

cis as well as *trans* pyrano tetrahydroquinolines were synthesized using a polymeric solid acid catalyst, polyaniline-*p*-toluenesulfonate (PANI–PTSA) salt, by conveniently controlling the reaction conditions. One-pot three component aza-Diels–Alder reactions involving aromatic aldehydes, aniline, and 3,4-dihydro-2*H*-pyran are effectively catalyzed by PANI–PTSA under solvent free condition at room temperature afford *cis* pyranoquinolines and the reaction carried out at 80 °C gave *trans* configuration. Whereas reactions involving 2,3-dihydrofuran instead of 3,4-dihydro-2*H*-pyran gave major *cis* furanoquinolines under solvent free conditions and 100% *cis* configuration with the use of solvents. This process conforms to the principles of 'green' chemistry by the usage of solvent free medium, easily handleable, recyclable and eco-friendly nature of the catalyst. In addition, this method provides simple work up procedure, short reaction time and use of easily synthesizable cheaper catalyst.

1. Introduction

Aza-Diels–Alder reactions provides a very important and most powerful method for rapidly constructing aza heterocyclics with nitrogen-containing six-membered ring compounds [1–6]. In recent years, increasing attention has been given to the synthesis of pyrano[3,2-*c*]- or furano[3,2-*c*] tetrahydroquinolines via three-component aza-Diels–Alder reactions of aryl aldehyde, aryl amine with cyclic enol ethers. Pyrano or furano quinoline derivatives are an important class of natural products and exhibit a wide spectrum of biological activities, such as antiallergic, anti-inflammatory, antipyretic, analgesic, antiplatelet, psychotropic, estrogenic activity anti-arrhythmic, immunosuppressive and anticancer properties [7–15]. In addition to this they are also used in pharmaceuticals [16].

The Imino Diels–Alder reactions with [4+2] cycloaddition reactions between *N*-arylimines (heterodienes) and enol ethers (dienophiles) under Lewis acid catalysis conditions have long been recognized as one of the most convenient methods for the synthesis of pyrano[3,2-*c*]- or furano[3,2-*c*]quinolines and have been explored for catalysts, scope and applications [17]. However, many Lewis acids cannot be utilized for the single-step coupling of aldehydes, amines and enol ethers because they will be decomposed or deactivated by the amines and water that exist during intermediate imine formation. Thus, more than stoichiometric amounts of the Lewis acids are required because the acids can be trapped by nitrogen of both the reactant and the product. Moreover some of the Lewis acids are not easily available or expensive, non-reusable, toxic and most of the imines are hygroscopic and unstable at high temperature. Thus a one-pot three component coupling protocol is highly desirable. Subsequently, one-pot procedures have been developed for this transformation with better results using various catalysts [18–25], and are reported in Table 1.

Normally, the process affords the products as a mixture of *cis* and *trans* isomers. Catalysts such as VCl_3 [18], PMA [19], SmI_2 [20] and $SbCl_3$–HAP [21] gave *trans* isomer as major product, whereas, Selectfluor™ [22], $GdCl_3$ [23], [bmim]BF_4 [24] and $Sm(OTf)_3$ [25] catalysts gave *cis* isomer as major product. SmI_2 catalyzed reaction gave major *trans* isomer (96% selectivity) at 50 °C in THF solvent and major *cis* isomer (77% selectivity) at 0 °C in solvent free conditions. 100% *trans* stereoselectivity was reported with the use of $SbCl_3$–HAP [21] catalyst in 2.5 h in acetonitrile solvent under reflux condition. It is noteworthy that acid catalyzed one-pot reactions affording the tetrahydroquinolines stereoselectively with particular preference for the *cis* isomers was a relatively unexplored area with limited number of literatures in the past.

Moreover, the drawbacks of some of these methods are: require strongly acidic conditions, use of hazardous catalysts, formation of mixtures of products, unsatisfactory yields, longer reaction times and use of expensive catalysts. Therefore, the development of mild,

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Table 1
Reports on the synthesis of pyrano[3,2-*c*]quinoline from aniline, benzaldehyde and dihydropyran using various catalysts.

Entry	Catalyst	Conditions	Time (h)	*trans:cis* (yield [%])	Recycle	Ref.
1	VCl_3	CH_3CN	2.5	80:20 (90)	N	[18]
2	PMA	Solvent free	3.5	90:10 (90)	N	[19]
3a	SmI_2	50 °C, THF	5	96:4 (93)	N	[20]
3b	SmI_2	0 °C, solvent free	24	23:77 (70)	N	[20]
4	$SbCl_3$–HAP	CH_3CN, reflux	2.5	100:0 (85)	Y	[21]
5	Selectfluor™	CH_3CN	3	15:85 (92)	N	[22]
6	$GdCl_3$	0 °C, THF	30	12:88 (80)	N	[23]
7	[bmim]BF_4	Solvent free	3	10:90 (91)	Y	[24]
8	$Sm(OTf)_3$	DCM	2.5	8:92 (90)	N	[25]
9a	PANI–PTSA	Solvent free	0.33	0:100 (78)	Y	[a]
9b	PANI–PTSA	Solvent free, 80 °C	0.33	100:0 (79)	Y	[a]

[a] Present work.

convenient and efficient procedures would extend the scope of this methodology to the synthesis of highly functionalized pyrano or furano quinolines.

In view of the emerging importance of polymer supported acid catalyst in organic synthesis [26–31], we herein report our results on polyaniline-*p*-toluenesulfonate salt catalyzed one-pot synthesis of aromatic aldehydes, aniline and cyclic enol ether for the stereoselective synthesis of *cis* or *trans* fused tetrahydroquinolines by controlling the reaction conditions.

2. Experimental

2.1. Instruments and characterization

Powder of PANI–PTSA salt was pressed into a disk of 13 mm diameter and about 1.5 mm thickness under a pressure of 400 MPa. Resistance of the pellet was measured by four probe method using 6220 constant current source and 2182 A voltmeter (Keithley, Cleveland, Ohio, USA). FT-IR spectra of PANI–PTSA salts were registered on a FT-IR spectrometer (Thermo Nicolet Nexus 670, USA) using the KBr pressed pellets technique. X-ray diffraction profiles for PANI–PTSA salt powders were obtained on a Siemens/D-500 X-ray diffractometer, USA using Cu Kα radiation, scan speed of 0.045°/min. Morphological studies of PANI–PTSA salt powders were performed using Hitachi 3000 N, Tokyo, Japan scanning electron microscope operating at 10 kV. The sample was mounted on a double-sided adhesive carbon disk and sputter-coated with a thin layer of gold to prevent sample from possible charging.

2.2. General procedure for preparation of PANI–PTSA salt

Polyaniline base was prepared by aqueous polymerization pathway as reported in literature [32]. Polyaniline base prepared above was stirred in 100 ml aqueous solution containing 0.2 M *p*-toluenesulfonic acid for 4 h, filtered, washed with excess water, finally with acetone and dried at 50 °C to a constant weight.

2.3. General experimental procedure for the synthesis of tetrahydro quinolines

In a typical experiment, mixture of aniline (1 mmol), benzaldehyde (1 mmol), cyclic enol ether (1.5 mmol) and PANI–PTSA (25 wt%, w.r.t. aldehyde), solvent (5 ml) or without solvent (solvent free condition) were stirred for 20–240 min and from room temperature to 100 °C. After the reaction, ethyl acetate was added to the reaction mixture, filtered and separated the PANI–PTSA catalyst. Organic layer was removed, washed with water, dried with anhydrous Na_2SO_4 and concentrated in vacuo. Crude products were purified by column chromatography. Products were authenticated with ^{1}H NMR spectra of known compounds. Diasteroselectivity of the products was determined from ^{1}H NMR spectra.

3. Results and discussion

3.1. Preparation and characterization of polyaniline catalyst

Polyaniline-*p*-toluenesulfonate salt (PANI–PTSA) [Scheme 1] was prepared via our earlier report [32] by oxidizing aniline using sodium persulfate oxidant in the presence of hydrochloric acid, followed by dedoping of polyaniline-hydrochloride salt (PANI–HCl) to polyaniline base (PANI) and subsequent doping with *p*-toluenesulfonic acid.

3.1.1. Conductivity of PANI–PTSA

Conductivity of the polyaniline salt measured by four probe method showed 1.1 S/cm, which is 12 orders of magnitude higher than that of PANI base (insulator level, $<10^{-12}$ S/cm) measured by two probe. Increase in conductivity result confirms that insulating PANI base is converted to doped PANI salt.

3.1.2. Infrared spectra of PANI–PTSA

The infrared spectrum of PANI base (Fig. 1a) showed major characteristic peaks at 3450, 2920, 2850, 1580, 1495, 1375, 1295, 1210,

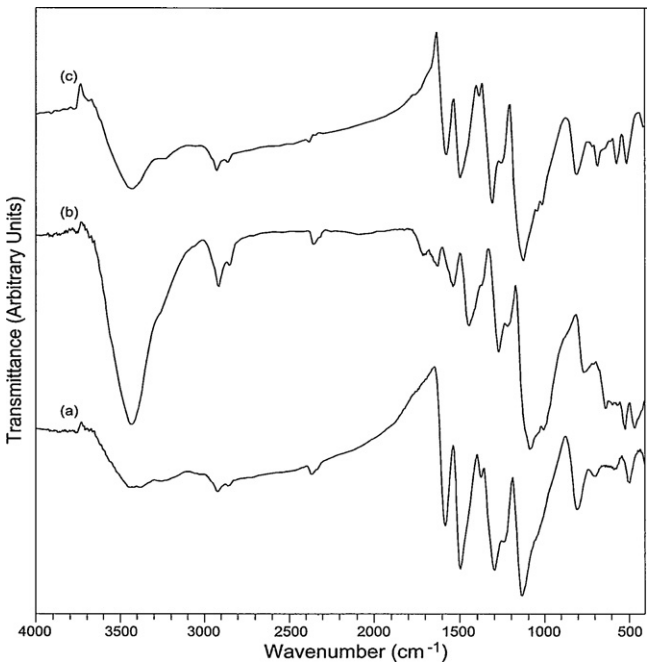
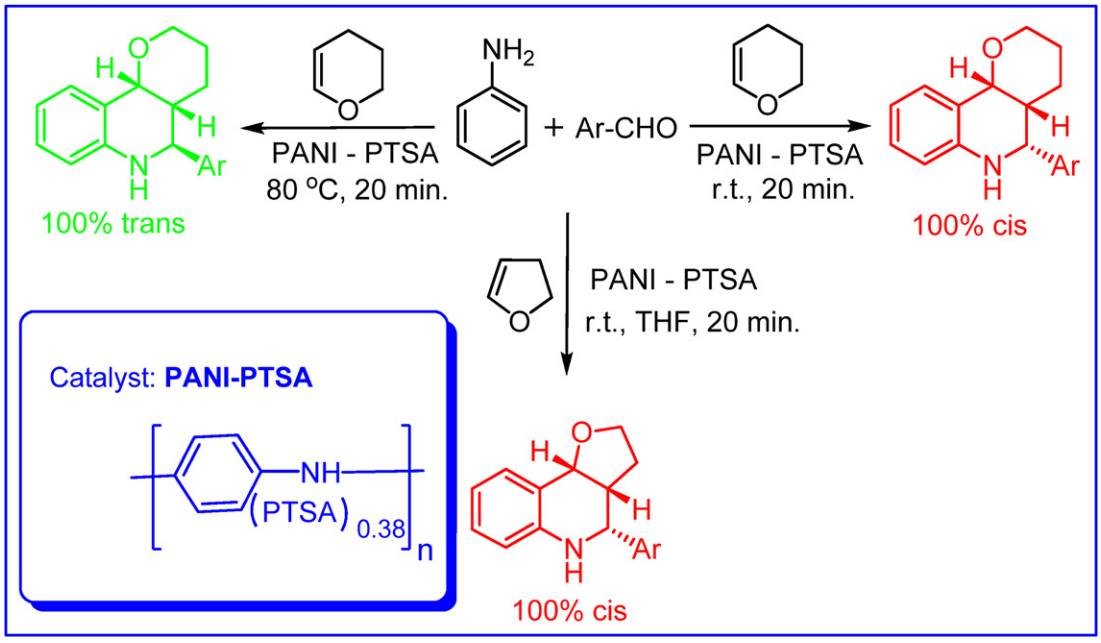


Fig. 1. Infrared spectra of (a) PANI-base, (b) PANI–PTSA and (c) recycled PANI–PTSA.



Scheme 1. Synthesis of *cis/trans* pyrano and *cis* furano tetrahydroquinolines using PANI–PTSA catalyst.

1135 and 810 cm^{-1}. A broad band at 3450 cm^{-1} assigned to the free N–H stretching vibration. The bands at 2920 and 2850 cm^{-1} are assigned to vibration associated with –NH part in $C_6H_4NH_2C_6H_4$ group or sum frequency. The ring stretching of quinoid and benzenoid form is observed at 1585 and 1480 cm^{-1}, respectively. The C–N stretching band of an aromatic amine appears at 1300 and 1225 cm^{-1}. Polyaniline shows a strong band at 1115 cm^{-1}, which has been explained as electronic like absorption of N=Q=N (where Q denotes quinoid ring). The C–H out-of-plane bending mode has been used as a key to identifying the type of substituted benzene.

The infrared spectrum of redoped PANI–PTSA salt is shown in Fig. 1b. The spectrum shows major peaks at 3440, 2920, 1585, 1480, 1300, 1215, 1115 and 805 cm^{-1}, which are similar to that of spectrum for PANI-base. In addition to these peaks, a peak appeared at 3225 cm^{-1} (Fig. 1b). This peak indicates that PANI base contains a dopant i.e., formation of PANI salt. Polyaniline salt shows peaks at around 1715, 1650, 1030 and 1015 cm^{-1} due to SO_3H group, indicating the presence of PTSA on polyaniline salt. These values are similar to the previously reported infrared spectrum of PANI–PTSA [32].

3.1.3. XRD patterns of polyaniline

X-ray diffraction pattern of PANI base (Fig. 2a) showed a broad peak around $2\theta = 19^\circ$, which is a characteristic peak of polyaniline base. X-ray diffraction profiles registered for PANI–PTSA (Fig. 2b) showed three broad peaks around $2\theta = 6.2^\circ$, 20° and 25° with corresponding *d*-spacing 14, 4.4 and 3.5 indicates the semi crystalline nature.

3.1.4. SEM and EDAX of polyaniline

Morphological structure of polyaniline samples were found out from scanning electron microscopy. PANI base showed agglomerated particles (Fig. 3), whereas, polyaniline salt showed agglomerated fibre like structure (Fig. 4). EDAX of PANI–PTSA indicates the presence of elements such as carbon, nitrogen and sulphur. This result supports that PANI salt contains PTSA dopant.

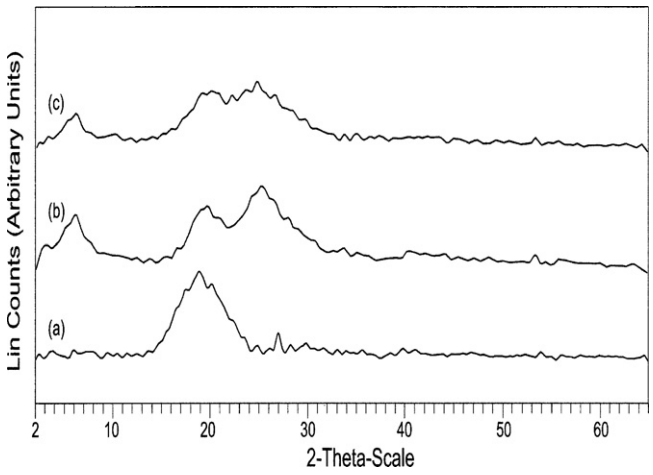


Fig. 2. X-ray diffraction patterns of (a) PANI-base, (b) PANI–PTSA and (c) recycled PANI–PTSA.

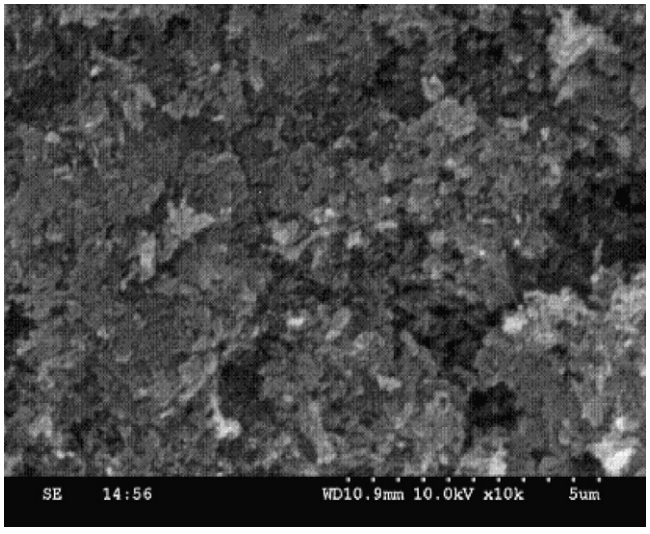


Fig. 3. SEM picture of PANI-base.

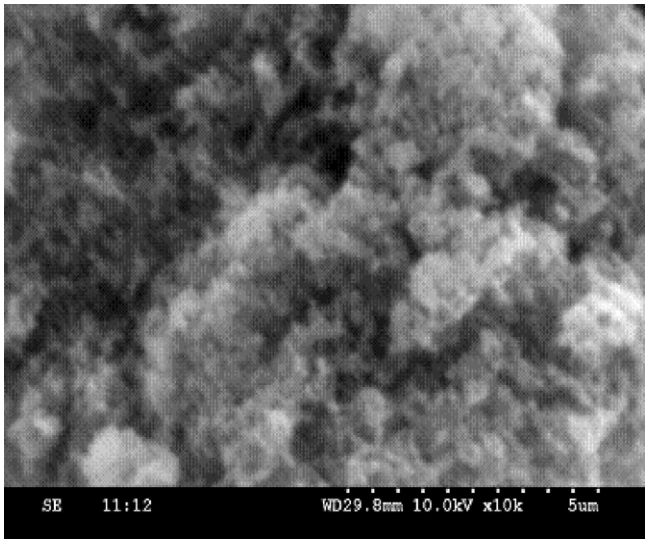


Fig. 4. SEM picture of PANI–PTSA.

3.2. Synthesis of tetrahydroquinolines using PANI–PTSA catalyst

In continuation of our work on the use of conjugated conducting polymers as a reusable catalysts for carrying out organic transformations [31], in this paper, we carried out stereoselective synthetic of tetrahydroquinolines by one-pot three component aza-Diels–Alder reactions involving aromatic aldehydes, aniline and 3,4-dihydro-2*H*-pyran or 2,3-dihydrofuran by employing PANI–PTSA as polymeric solid acid catalyst to afford *cis* or *trans* tetrahydroquinolines by controlling the synthesis procedure (Scheme 1).

3.3. Synthesis of pyrano[3,2-c] tetrahydroquinolines using PANI–PTSA catalyst

In a typical procedure, the reaction was carried out with benzaldehyde (1 mmol), aniline (1 mmol), 3,4-dihydro-2*H*-pyran (1.5 mmol), PANI–PTSA catalyst (25 wt% with respect to benzaldehyde) under solvent free condition at room temperature for 20 min., which gave *cis* pyranoquinoline in 78% yield (Table 2), whereas, at 80 °C gave *trans* configuration (79% yield). Higher temperature gave more of the thermodynamically stable *trans* products, while lower temperatures resulted in fast formation of the kinetically favored *cis* products. In order to optimize the catalyst amount, the reaction was carried under solvent free condition at room temperature with increasing amount of catalyst. The product (*cis* configuration) was obtained (78%) in 20 min with the use of 25 wt% or greater amount of catalyst.

Table 2
Solvent screening for the synthesis of pyrano[3,2-*c*]quinoline.[a]

Entry	Solvent	Temp	Time (h)	Isolated yield (%)	*trans:cis*
1	No Solvent	RT	0.33	78	0:100
2	No Solvent	80 °C	0.33	79	100:0
3	CH_3CN	RT	0.33	85	66:36
4	CH_3CN	Reflux	0.33	90	75:25
5	DCM	RT	0.33	86	0:100
6	THF	RT	0.33	87	0:100
7	C_2H_5OH	RT	4	65	63:37
8	CH_3OH	RT	4	45	41:59
9	H_2O	RT	12	ND[b]	ND[b]
10	H_2O	Reflux	4	62	83:17
11	DMSO	RT	4	54	84:16
12	C_6H_6	RT	4	75	88:12
13	$CHCl_3$	RT	4	59	0:100

[a] Aniline (1 mm), benzaldehyde (1 mm), 3,4-dihydro-2*H*-pyran (1.5 mm), solvent (5 ml), PANI–PTSA.
[b] not detected.

Table 3
Synthesis of pyrano[3,2-*c*]quinolines under solvent free condition.[a]

Entry	Ar	Room temp		80 °C	
		Isolated yield (%)	*trans:cis*	Isolated yield (%)	*trans: cis*
1	C_6H_5	78	0:100	79	100:0
2	4-ClC_6H_4	84	0:100	86	100:0
3	4-BrC_6H_4	82	0:100	85	100:0
4	4-FC_6H_4	84	0:100	83	100:0
5	3,4-$Cl_2C_6H_3$	78	0:100	82	90:10
6	4-$OCH_3C_6H_4$	83	0:100	88	100:0
7	2-Furyl	81	0:100	83	100:0
8	1-Napthyl	84	0:100	87	100:0
9	4-$CH_3C_6H_4$	88	0:100	90	100:0

[a] aniline (1 mm), aromatic aldehydes (1 mm), 3,4-dihydro-2*H*-pyran (1.5 mm), PANI–PTSA, reaction time-20 min.

In order to address the effect of solvent on this methodology, the standard reaction was carried out in various solvents (Table 2). Among the nine solvents used, chloroform, dichloromethane and tetrahydrofuran favored the *cis* product formation, while the solvents acetonitrile, dimethylsulfoxide, water, ethanol and benzene favored the formation of *trans* isomer. Solvent conditions played a major role in affecting the diastereoselectivity of the products. Higher yield of *cis* product (87%) was obtained with the use of tetrahydrofuran solvent.

The standard reaction under solvent free condition was further extended by changing the aromatic aldehydes and the results are reported in Table 3 (entries 1–9). All the crude products were authenticated by comparison with ^{1}H NMR spectra. Diastereoselectivity of the product was also found out from NMR. All the aldehydes gave *cis* product (yield 78–88%) when the reaction was carried out at room temperature and gave *trans* product at 80 °C (yield 78–90%) except that 3,4-dichloro benzaldehyde which gave major *trans* product.

3.4. Synthesis of furano[3,2-c] tetrahydroquinolines using PANI–PTSA catalyst

In order to check the versatility, aza-Diels–Alder reactions was also carried out by using 2,3-dihyrofuran instead of 3,4-dihydro-2*H*-pyran with PANI–PTSA catalyst (Table 4). The reaction carried out with 4-chloro benzaldehyde under solvent free condition at room temperature gave 100% *cis* selectivity and however at 80 °C, the reaction yielded *cis* isomer (72%) as the major product (Table 4, Entry 3). The reaction with benzaldehyde (Table 4, Entry 1) and 4-methoxy benzaldehyde (Table 4, Entry 2) carried out both at room temperature and 80 °C gave *cis* isomer as the major product.

In order to increase the performance, the reaction was carried out using solvents such as THF, DCM and $CHCl_3$ at room temperature. This reaction gave *cis* fused furanoquinoline with the yield

Table 4
Synthesis of furano[3,2-*c*]quinoline under solvent free condition.[a]

Entry	Ar	Room temp		80 °C	
		Isolated yield (%)	*trans:cis*	Isolated yield (%)	*trans: cis*
1	C_6H_5	78	28:72	79	33:67
2	4-$OCH_3C_6H_4$	81	17:83	84	22:78
3	4-ClC_6H_4	83	0:100	85	28:72

[a] Aniline (1 mm), aromatic aldehydes (1 mm), 2,3-dihydrofuran (1.5 mm), PANI–PTSA, reaction time 20 min.

Table 5
Synthesis of *cis* fused furano [3,2-*c*]quinoline.[a]

Entry	Ar	Isolated yield (%)	*trans:cis*
1	C_6H_5	78	0:100
2	2-Furyl	78	0:100
3	4-FC_6H_4	81	0:100
4	4-ClC_6H_4	76	0:100
5	4-BrC_6H_4	80	0:100
6	4-$CH_3C_6H_4$	84	0:100
7	3,4-$Cl_2C_6H_3$	79	0:100
8	2-Cl, 5-$NO_2C_6H_3$	69	0:100

[a] Aniline (1 mm), aromatic aldehydes (1 mm), 2,3-dihydrofuran (1.5 mm), THF solvent (5 ml), PANI–PTSA, reaction time 20 min.

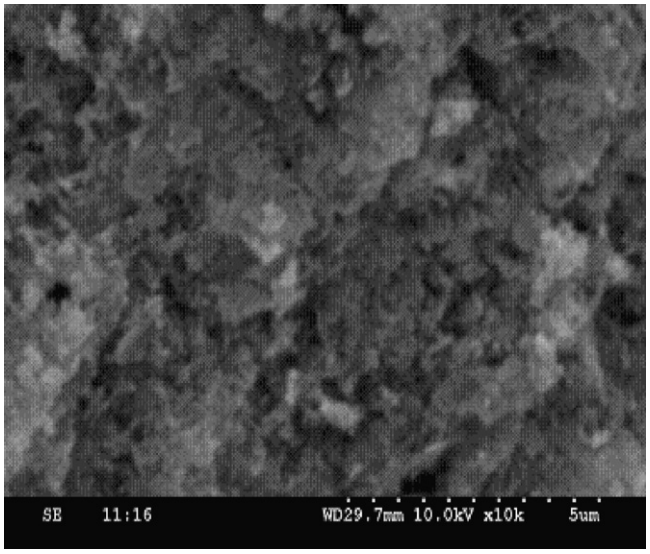


Fig. 5. SEM picture of recycled PANI–PTSA.

of 78, 75 and 61% when the use of THF, DCM and $CHCl_3$ as solvent, respectively.

The reaction was extended further using various aromatic aldehydes in THF solvent and the results are reported in Table 5. In all the cases, the reactions proceeded smoothly and gave *cis* products in very good yield.

3.5. Recyclability

Reusability of the PANI–PTSA was verified for the standard reaction of aniline, benzaldehyde with 3,4-dihydro-2*H*-pyran under solvent free condition at room temperature for 20 min. The reaction gave *cis* isomer with 78% yield. Recyclability was checked by separating the catalyst from the reaction mixture by filtration and carrying the standard reaction again using the separated catalyst. This procedure was used for four reactions and the *cis* isomer was obtained in 100, 99, 100 and 99 selectivity with the yield of 82, 77, 79 and 81%, respectively. After the recyclability experiment, the catalyst was analyzed by infrared (Fig. 1c), X-ray diffraction (Fig. 2c) and SEM (Fig. 5) analyses and similar results were obtained before the reaction (Figs. 1b, 2b and 4, respectively). The above results indicate that PANI–PTSA catalyst show good recyclability.

4. Conclusion

In this paper, polyaniline-*p*-toluenesulfonate salt is demonstrated as reusable polymer based solid acid catalyst for the controllable stereoselective synthesis of *cis* as well as *trans* pyrano and also *cis* furano tetrahydroquinolines in short reaction time (20 min). This methodology is amenable to scale up.

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