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Effects of microwave radiation on one-step oxidation of benzene to phenol with nitrous oxide over Fe-ZSM-5 catalyst

S. Gopalakrishnan, J. Münch, R. Herrmann, W. Schwieger*

Friedrich-Alexander Universität Erlangen-Nürnberg, Lehrstuhl für Chemische Reaktionstechnik, Egerlandstrasse 3, 91058 Erlangen, Germany

Abstract

Direct oxidation of benzene to phenol over Fe-ZSM-5 catalyst was chosen as a model reaction to characterize the influence of microwave irradiation on ad-/desorption processes during heterogeneously catalyzed reactions. The direct oxidation of benzene was investigated under different reaction conditions (temperature, residence time, reactant-concentrations) and microwave power in a specially designed reactor. The results obtained by additional microwave radiation were compared to those obtained by conventional heating under similar reaction conditions. In addition, ex situ TG-MS analyses were also conducted to study the sorption behaviour. Extensive investigations were carried out to determine adsorption–desorption effects (benzene, phenol and a mixture of both) and the coking behaviour under microwave conditions. Microwave induces a trend towards increased phenol selectivity and relative deactivation during the reaction. However, there was no conclusive improvement in the performance was obtained by means of microwave heating.

Keywords: Microwave; Benzene oxidation; Nitrous oxide; Phenol; Zeolite; Selective desorption

1. Introduction

The concept of using microwave as an energy source for chemical reactions involving solid catalysts has been investigated intensively in recent years [1,2]. These studies show that in some cases the reaction rates are enhanced while in others the product selectivity changes when microwave radiation is used instead of conventional heating. Whether this is due to faster and selective dielectric heating provided by microwave or due to any specific microwave effect (e.g.: specific bond activation), is a matter of debate [3–7].

An advantage of microwave heating compared to other heating methods is that any substance with high dielectric loss factor (ability of the substance to absorb the electromagnetic energy) can couple easily with microwave radiation and gets heated. But substances with low dielectric losses remain nearly unheated under microwave radiation. This effect can also be used to influence desorption processes on adsorbents. If an adsorbent (catalyst) has a low dielectric loss factor and is loaded with two species, namely, non-polar and polar, the latter can be selectively desorbed by microwave radiation [8].

In this work direct oxidation of benzene to phenol on Fe-ZSM-5 catalyst with N_2O as an oxidant is used as a model reaction to determine the influence of microwave irradiation on catalytic reactions. The choice of the model reaction is not only due to the economic and scientific importance of the reaction but also due to the fact that this reaction has been investigated extensively in the past 20 years [9,10]. The biggest problem of this reaction is the short life time of the catalyst due to coking and by-product formation. Recently, it has been reported that the strong phenol adsorption on the catalyst is supposed to be the reason for the coking process [11]. A solution to that problem is rather modifying the catalyst or optimizing the process. A third possibility, on which this work is based, is to selectively desorb phenol by microwave radiation. Most importantly, unlike phenol, the reactants (benzene and nitrous oxide) and the catalyst have low dielectric loss factors. Hence microwave should selectively heat phenol and thus facilitate selective desorption. This effect (selective desorption) should reduce coke formation which in turn will increase phenol selectivity and the catalyst lifespan. A schematic representation of selective desorption of phenol is illustrated in Fig. 1. In this work we report about our investigation on the principle of selective desorption by microwave in heterogeneous catalysis. Therefore, the conventional and the microwave assisted process are compared under identical conditions.

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* Corresponding author. Tel.: +49 9131 852 8910; fax: +49 9131 852 7421.
E-mail address: wilhelm.schwieger@rzmail.uni-erlangen.de (W. Schwieger).

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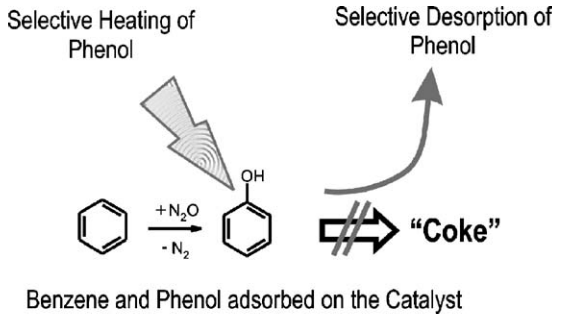

Fig. 1. Reaction scheme describing the idea of microwave selective desorption of phenol.

2. Experimental

2.1. Experimental setup

The laboratory scale experimental setup consists of a packed bed quartz glass reactor and a multimode microwave oven (Ethos 1600; MWS). The setup allows combined dielectric and conventional heating with an exact temperature control (IR-Pyrometer) and minimal temperature gradients within the catalyst bed. A detailed description of the experimental setup is given elsewhere [12].

Benzene was dosed with a HPLC pump (K-120; Knauer) and evaporated with N_2 as carrier gas. Nitrous oxide and nitrogen were dosed with mass flow controllers. An online gas chromatograph (VARIAN 3600) was used for the analysis of reactants and products. The GC was equipped with FID and TCD detectors and fused silica column of type HP-5 from the manufacturer Hewlett Packard (length 25 m; Ø 0.53 mm). Methane was used as the internal standard for the product analysis (GC) and measurements were taken for every 8 min over a period of 2 h. The benzene conversion was calculated from the molar ratio of converted amount of benzene to its initial amount. The yield of phenol was calculated from the obtained molar amount of phenol. The selectivity of phenol is defined as the molar ratio of the phenol obtained relative to benzene conversion. Modified residence time (τ_{mod}) was calculated as the ratio of weight of the catalyst to the molar flow rate of the reactant gas mixture. The catalyst was regenerated to enable its continuous usage after each reaction. Complete regeneration was achieved by heating the catalyst to 530 °C using an oxygen/nitrogen mixture for one hour in order to restore the initial activity. The temperature was then decreased to the desired reaction temperature under a flow of nitrogen (200 ml_N/min) for 1 h. Prior to each reaction, by-pass measurements were taken to determine the reactant concentration in the feed gas. Influence of various parameters such as temperature (300–400 °C), residence times (90 and 120 g min/mol) and microwave power settings (100 W and 200 W) were investigated.

2.2. Catalyst preparation

Fe-form of zeolite ZSM-5 ($SiO_2/Al_2O_3 = 92$) was used as catalyst throughout this work. This Fe-ZSM-5 catalyst contained 1.16 wt.% of Fe. The catalyst powder was calcined at 900 °C. The catalyst powder was pressed into pellets before being crushed and sieved to obtain a particle size of 1–1.2 mm. One gram of catalyst was diluted with 20 ml of quartz glass beads (0.8–1.0 mm particle size) and filled in the reactor. Preliminary heat up experiments showed that this catalyst and quartz mixture is transparent to microwave radiation and it will not be heated by microwave.

2.3. Catalyst characterisation

Thermogravimetry coupled with mass spectroscopy (TG-MS) was employed to identify the adsorbed species and to determine the temperature required to desorb each species from the catalyst surface. The used TG equipment was of type SDT 2960 (TA Instruments). A quadrupole mass spectroscope of type Thermostar 2000 (BALZERS) was connected to the outlet of the TG furnace.

2.4. Experimental procedure

The aim of this work was to achieve selective desorption of phenol as well as to determine the influence of microwave on benzene oxidation reaction. Systematic investigations were carried out in order to realize this aim. Firstly, the influence of microwave on N_2O decomposition was tested as it is considered to be the vital part in this reaction [9]. Secondly, the influence of microwave on the benzene oxidation reaction was investigated by comparing the experimental results obtained by using microwave radiation with the one obtained by conventional heating. Finally, TG-MS analysis of ex situ loaded samples and spent catalysts from the reactions were conducted. This was done to get an insight into the effect of microwave on adsorption/desorption properties of the reactants and the product.

It is important to know the influence of microwave on the N_2O decomposition reaction before applying microwave in the benzene oxidation reaction. N_2O decomposition experiments were carried out in the same experimental setup as the benzene oxidation experiments. N_2O was detected by an infra red N_2O analyser of the type URAS 10E (Hartmann & Braun). This detector was detached during the benzene oxidation experiments. By keeping the residence time constant, N_2O decomposition was conducted with conventional heating by varying the bed temperature (300–500 °C). Each experiment was repeated with additional 120 W of microwave power.

The investigation of one-step benzene oxidation reaction includes observing benzene conversion (X), phenol yield (Y), phenol selectivity (S), benzoquinone selectivity (S) and relative deactivation (ΔX) with time on stream (TOS). All these experiments were performed with conventional as well as with combined conventional/microwave heating. Reason for this is that a catalyst transparent for microwave radiation cannot be heated to the desired reaction temperature by microwave radiation alone. This mixed energy input is called as "microwave assisted reactions/experiments". It should be noted that during microwave assisted experiments the catalyst bed temperature

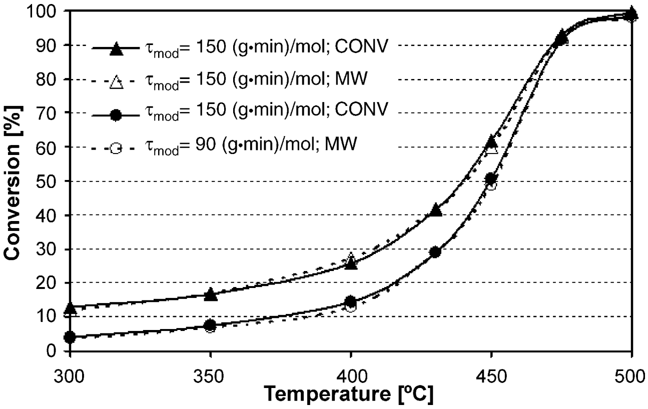

Fig. 2. Activity of Fe-ZSM-5 catalyst in the N_2O decomposition as a function of temperature; Comparison of 120 W MW assisted reaction (dotted lines) with conventional reaction (solid lines) at two different modified residence times 120 and 150 g min/mol.

was maintained constant by conventional heating and not by microwave power (MW) [9].

A set of reactions was carried out by keeping the modified residence time (τ_{mod}) constant and by varying the key parameters such as reaction temperature, feed ratio, and microwave power in order to determine the effect of microwave radiation on the benzene oxidation reaction. Each experiment was conducted with conventional heating as well as with microwave of power 100 and 200 W. This 'all-in-one' conventional and microwave investigation provides best conditions for comparison of the results.

3. Results and discussion

3.1. N_2O decomposition

The results of N_2O decomposition over Fe-ZSM-5 with conventional heating and microwave heating are plotted in Fig. 2. The conversion of N_2O was compared for the modified residence time of 90 and 150 g min/mol. Under both conditions, N_2O conversion increases with temperature. But this increase in conversion was found to be higher at temperatures beyond 400 °C. Till 400 °C, the maximum conversion was observed to be 25.8%. Conversion reaches 100% at 500 °C. The good fit of the graphs for conventional and microwave assisted experiments indicate that microwave has no influence on N_2O decomposition over Fe-ZSM-5. This is a precondition for the investigation of microwave assisted selective phenol desorption in which no microwave enhancement of the N_2O decomposition has to be taken into account.

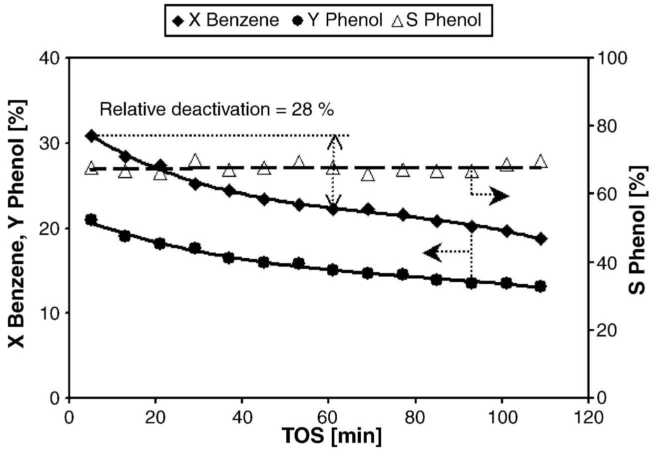

Fig. 3. Benzene conversion, phenol yield and phenol selectivity as a function of catalyst time on stream (TOS) [$\tau_{\mathrm{mod}} = 90$ g min/mol, reactor temperature = 400 °C, molar ratio of reactants N_2O:benzene = 1:1].

3.2. Benzene oxidation

Fig. 3 displays the benzene conversion, phenol yield and the selectivity of phenol as a function of time on stream with a stoichiometric feed ratio (1:1) at τ_{mod} 90 g min/mol and at 400 °C. Benzene conversion after starting the reaction (5 min TOS) was 31% at this condition. The relative deactivation (ΔX) between 5 and 61 min TOS was 28%. And the phenol selectivity (based on benzene) lay at around 67% and it was approximately constant for a period of 100 min TOS. Fig. 4 compares the influence of temperature as well as the influence of microwave on catalytic behaviour.

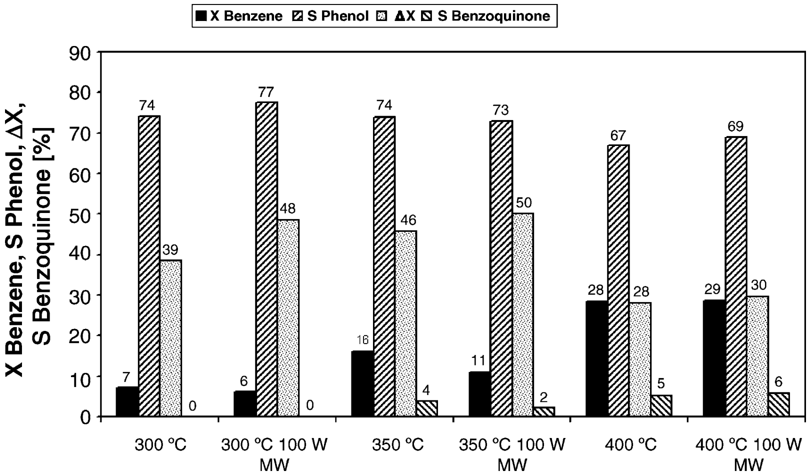

Fig. 4. Catalytic behaviour of the Fe-ZSM-5 with respect to temperature and microwave power [$\tau_{\mathrm{mod}} = 90$ g min/mol, N_2O:benzene = 1:1, TOS = 13 min].

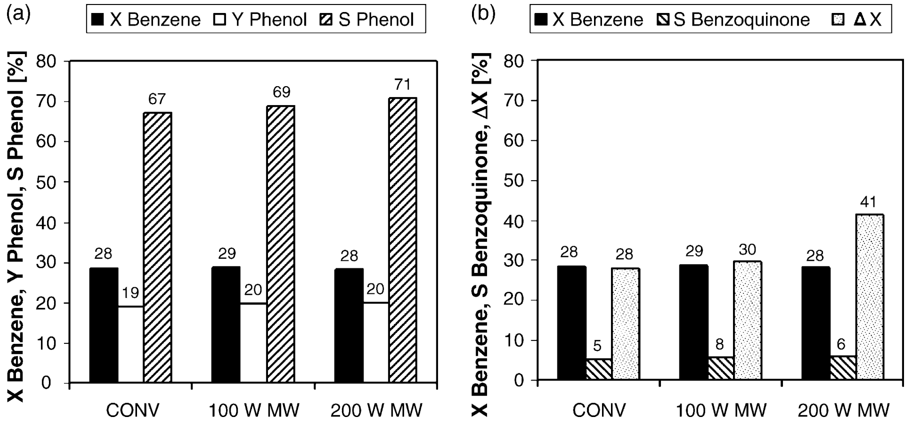

Fig. 5. Catalytic performance of the Fe-ZSM-5 with respect to microwave power [$\tau_{mod} = 90$ g min/mol, $T = 400$ °C, N_2O:benzene = 1:1, TOS = 13 min].

3.2.1. Conventional reactions

Considering the reactions without microwave one has to recognize two temperature regions which show different behaviour. In the low temperature region (300–350 °C) an increase in temperature increases the benzene conversion from 7 to 16% and the change in phenol selectivity was less (around 74% after 13 min TOS). A further raise in temperature enhances the benzene conversion to 28%. However, it was accompanied by a clear drop in selectivity to 67% at 400 °C. Only benzoquinone was formed in significant amount as volatile by-product in the investigated temperature range (300–400 °C). The selectivity of benzoquinone (related to benzene) increased with an increase in the temperature. The loss in phenol selectivity may be due to the formation of the by-product benzoquinone and coke. This is in accordance with the literature that higher temperature decreases phenol selectivity as it favours the by-product and coke formation [13].

3.2.2. Microwave assisted reactions

Experimental results of microwave assisted reactions were compared with those obtained by conventional heating (Fig. 4). It is reported that the strong adsorption of phenol is responsible for the undesired further oxidation and coking of the catalyst [11]. If the microwave enhances selective desorption of phenol, one would expect the following effects in the presence of this radiation:

1. an increase in the phenol selectivity,
2. a reduction in the relative deactivation (ΔX), and
3. suppression of consecutive reaction and by-product formation (selectivity towards benzoquinone should be less).

But the experimental observations are not completely in agreement with the expectations. The additional microwave radiation seems to have less influence on the phenol selectivity (related to benzene) in the investigated temperature range. And microwave seems to lead to reduced benzene conversion and to faster deactivation. At the reaction temperature 300 °C, there was an increase in phenol selectivity from 74 to 77% in the presence of microwave. But the relative deactivation of the catalyst increases by 9% under microwave conditions, which is not supposed to happen if microwave favours phenol desorption. The effect of microwave radiation seems to be well pronounced in the low temperature ranges.

At 350 °C, no change in phenol selectivity was observed under microwave conditions. However, there was about 4% increase in the relative deactivation with microwave. At 400 °C, there was very less difference between the conventional and microwave assisted reaction. At this temperature, phenol selectivity increased by 2% in the presence of microwave. On the contrary, the relative deactivation also increased by 2%, which is not supposed if microwave promotes selective desorption.

It should be noted that in the presence of microwave the benzoquinone selectivity (related to benzene) was reduced to 2% from 4% at 350 °C. But at 400 °C an increase from 5 to 6% was observed in the presence of microwave. All those small differences are obviously only weak hints for well based conclusions. Nevertheless a change within the reaction parameters between 350 and 400 °C has to be recognized.

Effects of irradiation during a reaction should reflect the intensity of the radiation. Hence, two different microwave powers (100 and 200 W) were applied and the resulting graphs are shown in Fig. 5. It was expected that the increase in microwave power should increase the phenol selectivity. There was a tendency towards higher phenol yield as well as enhanced phenol selectivity with more powerful microwave (Fig. 5a). However, the differences between absolute values lay only within 2%. The relative deactivation (Fig. 5b) increased about 11% when the microwave power was changed from 100 to 200 W. A trend towards deactivation has also to be noticed with a raise in microwave power. It should be noted that the differences were very less and lay in the precision limit of the experiment (analytical equipment).

Summarising the experimental results at the residence time 90 g min/mol, irrespective of the trend towards higher yield of phenol, microwave also seemed to accelerate the deactivation process of the catalyst. These contradictory results could be caused by a superposition of different effects like low phenol concentration on the catalyst surface preventing sufficient microwave heating and temporal discrepancies between activation time for microwave phenol desorption and residence time.

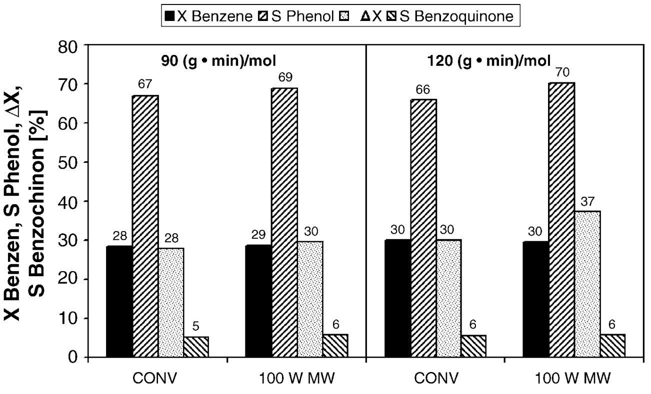

Fig. 6. Comparison of catalytic behaviour of Fe-ZSM-5 at two different residence times [$T = 400\,^{\circ}C$, N_2O:benzene = 1:1, TOS = 13 min].

3.2.3. *Variation of residence time*

A higher modified residence time of 120 g min/mol shall help render a clear microwave effect as the contact time of phenol with catalyst as well as with microwave increases. For that reason all experiments conducted for the residence time 90 g min/mol were repeated for the residence time 120 g min/mol.

Comparison of catalytic behaviour at 90 and 120 g min/mol residence time and the effect of microwave are given in Fig. 6. Around 2% increase in the benzene conversion and a slight decrease in phenol selectivity by 1% at residence time 120 g min/mol compared to 90 g min/mol was found. The benzoquinone selectivity did not change with residence time. Comparing conventionally heated experiments conducted at 120 g min/mol residence time with the corresponding microwave assisted reactions, phenol selectivity and relative deactivation were higher again with microwave radiation. But here this effect is well pronounced. Under microwave radiation, phenol selectivity raises from 66 to 70% while the relative deactivation went up from 30 to 37%. On the other hand benzene conversion did not change significantly under microwave radiation (less than 0.5%).

By summarising the results a trend towards a higher phenol selectivity and relative deactivation was observed using microwave radiation. These observations were partly opposite to our expectations, because microwave radiation was assumed to suppress the deactivation rate by increasing the phenol selectivity.

3.3. *TG-MS analysis*

As described above some differences were observed between microwave assisted and conventional benzene oxidation reactions within the investigated experimental conditions. These observations have opened up the following questions:

- Is phenol solely responsible for catalyst deactivation?
- Is phenol desorption a determining factor?
- How strong is the interaction of microwave with the adsorbed species?
- Is it possible to selectively desorb phenol from the catalyst surface by microwave when no reaction is taking place?

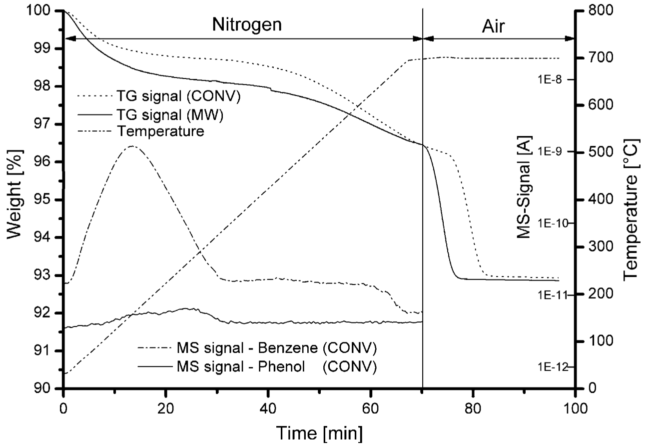

Fig. 7. TG-MS analysis of two Fe-ZSM-5 catalyst samples from benzene oxidation reaction [$T = 400\,^{\circ}C$, $\tau_{mod} = 90$ g min/mol, benzen:N_2O = 1:1 after 2 h TOS; CONV: without microwave; MW: 120 W microwave radiation].

For this purpose deactivated catalyst from the benzene oxidation reaction was analysed by TG-MS-measurements. Furthermore, fresh catalyst samples were loaded with benzene, phenol and a mixture of both substances. The catalyst samples were loaded with the desired substances and divided into two parts. One part was purged (N_2 pre-treatment) with 100 ml$_N$/min N_2 for 2 h. The other part was purged with 100 ml$_N$/min N_2 with additional microwave (400 W). These pre-treated samples were subjected to (TG-MS) analysis. It should be noted that the mass to charge ratio of 78 and 94 were used in the MS to detect benzene and phenol, respectively.

3.3.1. *Spent catalyst*

Fig. 7 compares the TG weight loss (black curves, left *Y*-axis) of the deactivated catalyst samples (spent catalyst) that were obtained from benzene oxidation reaction. One sample (CONV) was from a reaction conducted with conventional heating and the other (MW) was from a reaction with combined microwave heating of 120 W. All other reaction conditions were the same for both catalyst samples. The reactions were stopped after 2 h and the catalyst was purged for 3 min with N_2 (200 ml$_N$/min) in order to remove the reaction mixture from the reactor. After this deactivation, the samples were subjected to a temperature programmed desorption by heating the sample from room temperature to 700 °C at a ramp rate of 10 °C/min in flowing N_2. The carrier gas was switched to air for about 30 min at once the sample reached 700 °C. This temperature programme can also be seen in the graph.

Comparing the TG-curves, the catalyst from microwave assisted reaction (MW) showed a higher weight loss at the low temperature region (<180 °C) than that of the sample from conventionally heated reaction (CONV). Benzene was detected in the temperature range between 80 and 250 °C (maximum at 158 °C). Phenol was detected between 180 and 350 °C. From 450 °C onwards, the sample from the conventionally heated experiment (CONV) showed higher weight loss than that of the catalyst from microwave assisted reaction (MW). This TG-measurement indicated a higher amount of loosely adsorbed

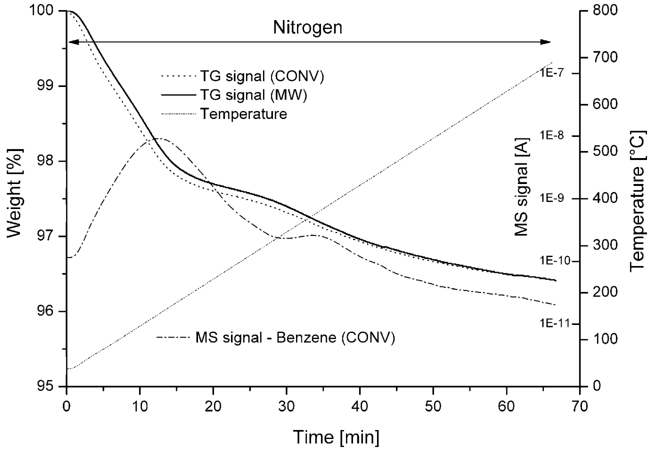

Fig. 8. TG-MS analysis of benzene loaded Fe-ZSM-5 catalyst samples [loading conditions: partial pressure benzene = 6450 Pa, T = 200 °C, 2 h; CONV: sample purged with N_2; MW: sample purged with N_2 and 400 W of microwave].

species present on the catalyst used in the microwave assisted reaction (MW). But at the same time species were stronger adsorbed on the catalyst from conventional reaction, causing an identical weightloss of about 3.5% at 700 °C for both samples. The amount of hard coke [14] which was determined by burning the coke with air at 700 °C resulted in about 3.6% for both samples.

These results show that microwave seems to have an influence on the kind of adsorbed substances and/or the strength of adsorption of different species on the catalyst surface, but not on the total amount of adsorbed species. Another interesting thing is that phenol desorption was found to take place predominantly at temperatures less than 300 °C in both the catalyst samples. This contradicts the argumentation that phenol leads to coking during benzene oxidation reaction due to its stronger adsorption.

3.3.2. Benzene loading

The catalyst was loaded with benzene at a partial pressure of 6450 Pa at 200 °C for 2 h. There was no change in colour of the catalyst after benzene loading. Fig. 8 shows the comparison of benzene loaded catalyst with N_2 (CONV) and additional microwave pre-treatment (MW). Both catalyst samples showed an identical weight loss of 3.6% at 700 °C. Two benzene peaks were found at 150 and 350 °C, respectively. This indicates the existence of two different adsorption states of benzene on the catalyst. No hard coke was detected.

3.3.3. Phenol loading

The catalyst was loaded with phenol at a partial pressure of 23,750 Pa at 200 °C for 2 h. The colour of the catalyst changed to grey during this procedure. Fig. 9, shows the TG weight loss of the phenol loaded catalyst. In N_2 up to 700 °C the microwave pre-treated sample (MW) showed a weight loss of 6.6% whereas conventionally pre-treated sample (CONV) showed a weight loss of 7.6%. Considering the MS signal it can be recognized that the phenol desorption takes place over a huge temperature range from 80 to 550 °C with a maximum at around 175 °C.

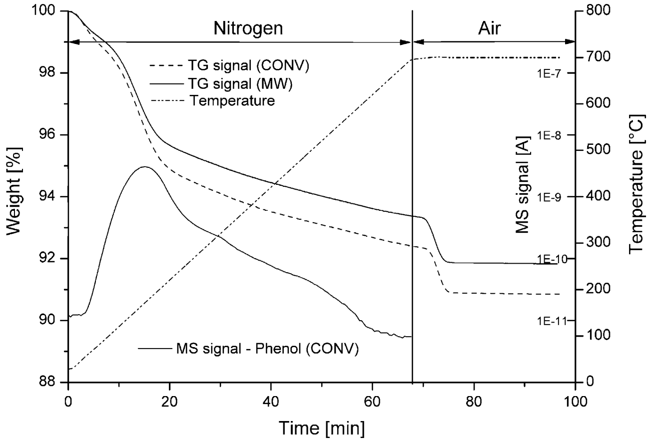

Fig. 9. TG-MS analysis of phenol loaded Fe-ZSM-5 catalyst samples [loading conditions: partial pressure of phenol = 23,750 Pa, 200 °C, 2 h; CONV: sample purged with N_2; MW: sample purged with N_2 and 400 W of microwave].

A strong deviation in weight loss between the two samples was found in the low temperature range up to 280 °C. This indicates that loosely bound phenol was desorbed from the catalyst during the pre-treatment with microwave. Both samples contained the same amount of hard coke (around 1.5%). Probably this might be the coke formed during the loading procedure at 200 °C (grey coloured catalyst after loading). These findings support that phenol is the coke precursor and also that coking takes place even without undergoing the hydroxylation reaction.

3.3.4. Mixture loading

One more catalyst sample was loaded with a mixture of phenol and benzene and pre-treated in the same way like above (Fig. 10). The two samples showed no deviations in weight change up to 180 °C. At higher temperature, the N_2 pre-treated catalyst sample (CONV) exhibited a higher weight loss up to 700 °C with N_2 as carrier gas than the microwave pre-treated sample (CONV: 6.3%; MW: 5.7%). The MS signal confirms that this weight loss was due to phenol desorption (maximum phenol desorption took place at around 180 °C while the max-

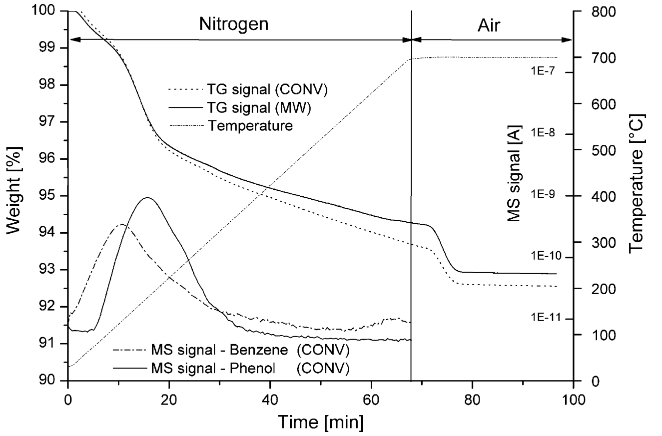

Fig. 10. TG-MS analysis of Fe-ZSM-5 loaded with the mixture of benzene and phenol [loading conditions: partial pressure of benzene = 25,690 Pa; Partial pressure of phenol = 26010 Pa; CONV: sample purged with N_2; MW: sample purged with N_2 and 400 W of microwave].

imum of the benzene desorption was found at around 130 °C). This verifies that microwave can selectively desorb phenol, but has no influence on benzene.

When the TG carrier gas was changed from N_2 to air, a drastic weight loss was noted which was due to burning off the hard coke. The proportion of hard coke in the microwave treated sample (MW) was 1.3% whereas CONV just showed a hard coke fraction of 1.1%. From the result of the previous loading experiment that the coke formation occurred during the phenol loading process, this difference in hard coke amount must have been caused by the coking reaction. The TG-MS investigation of the catalyst loaded with a mixture of benzene and phenol proves that microwave has the ability to selectively desorb phenol, while benzene is not influenced.

4. Conclusion

The differences between the microwave assisted and the conventionally heated one-step oxidation of benzene to phenol over Fe-ZSM-5 with N_2O were found to be small. A trend towards increasing relative deactivation and higher phenol selectivity under microwave radiation was observed. This effect can be intensified by increased residence time or microwave power. However, there was no conclusive improvement in the performance was achieved by means of microwave heating.

Considering the TG-MS results, we conclude that microwave has the ability to heat adsorbed phenol on solid catalysts selectively. TG-MS results indicate that there are strongly as well as loosely adsorbed phenol molecules present on the catalyst surface. Due to the selective heating of microwave, in particular loosely adsorbed phenol was desorbed. This effectuates an increase in phenol selectivity. On the other hand the enhancement in the relative deactivation is attributed to the microwave selective heating of strongly adsorbed phenol molecules which cannot be desorbed and the reaction towards coke is accelerated due to the additional heating.

However, the TG-MS analysis of the catalyst samples from reaction (spent catalysts) prove that the phenol desorption is not crucial for the deactivation during benzene oxidation. The temperature needed to desorb the essential amount of phenol from the catalyst was found to be less than 300 °C. Since the oxidation of benzene with N_2O was carried out at higher temperatures (350 and 400 °C), it cannot be expected, that the microwave has a significant effect on phenol desorption. Samples loaded with phenol at 200 °C showed coke formation at temperatures lower than those necessary for the oxidation. This proves that phenol is the coke precursor, and that the "reaction" towards coke takes place more easily than the oxidation. To run the investigated process efficiently a catalyst which accelerates the direct oxidation more than the coke formation from the product phenol is required. In such a case microwave would be a benefit to keep the catalyst active by selective phenol desorption.

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